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Proceedings of the

FIFTH
COTTON UTILIZATION RESEARCH
CONFERENCE

Held at New Orleans, Louisiana

May 6 and 7, 1965

Agricultural Research Service

UNITED STATES DEPARTMENT OF AGRICULTURE

PREFACE

The Cotton Utilization Research Conference is sponsored annually by the Southern Utilization Research and Development Division to bring the results of recent studies in the areas of mechanical processing, chemical modification, and fundamental research on cotton, to the attention of both executive and research personnel in the textile and allied industries.

This Conference was held May 6 and 7, 1965, at the Fontainebleau Motor Hotel in New Orleans, Louisiana. Serving as Honorary Chairman was George S. Buck, Jr., of the National Cotton Council of America, and as General Chairman, J. Lindsay Dexter, of the Pepperell Manufacturing Company. The program was developed by B. H. Wojcik, Assistant Director, under the direction of C. H. Fisher, Director, in cooperation with staff members, and advisers representing the cotton industry.

These proceedings report in summary the statements presented by the various speakers during the conference and give an account of the discussions following.

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PROCEEDINGS OF THE FIFTH COTTON UTILIZATION RESEARCH CONFERENCE

Held at New Orleans, Louisiana, May 6 - 7, 1965

WELCOME

by
C. H. Fisher, Director
Southern Utilization Research and Development Division

It is my privilege and honor to say "greetings and welcome" at the beginning of our Fifth Cotton Utilization Research Conference. I hope and think that the Conference will be successful and rewarding. Certainly it will serve a useful purpose for members of the Division. We are complimented and grateful because we have such a splendid audience in both quality and number and we are pleased with your interest and participation.

The principal purpose of these annual Cotton Conferences is to report our cotton research promptly and to receive questions and comments about our research. We want our research results to prove useful as soon as possible. We also want your comments, particularly those that will help us improve our program. We are glad to have comments not only at this meeting, but also during the entire year. We invite you to telephone, write, or visit us to get information about our research and to pass on any comments you may wish to make.

During the past year there was one major change in our cotton research. Because of the durably pressed garment development and the need to improve wearlife, we increased emphasis, starting last fall, on research in this area--primarily to improve the abrasion resistance of all-cotton garments. In this connection, I am proud of our cotton researchers for two reasons. First, they had sufficient flexibility to change their work, and second, they were able in a short time to find several promising approaches and to improve cotton's competitive position. The results of this work will be presented Friday afternoon.

I wish to mention one aspect of our research, namely, our close cooperation with industrial and other organizations. This cooperation extends to all stages of our activities: First, in the planning stage, as exemplified by the fall meeting with our cotton research advisers; second, in the execution and evaluation of research, the evaluation is the purpose of our Spring Cotton Advisers; and third, in effective cooperation in the dissemination of research information and activities to have our results adopted by industry. This cooperation and help are most valuable to the Southern Division.

We express gratitude to the many persons contributing to the success of this Conference. These include: Honorary Chairman, Dr. George Buck; General Chairman, Mr. Lindsay Dexter; the Session Chairmen; the Program Committee; Dr. Wojcik, Mr. Persell, Mr. Bourdette, and their staffs; the speakers; the scientists and engineers doing the research; Messrs. Larry Heffner and Bill Martin for assistance in information dissemination and industrial development; and our splendid audience.

OPENING REMARKS

by
George S. Buck, Jr.
National Cotton Council of America
Memphis, Tennessee

Dr. Fisher assured me when he asked me to serve as honorary chairman of the Fifth Cotton Utilization Conference that the duties would be practically nonexistent. In several subsequent conversations he continued to emphasize the same point, that I would really have nothing to do. Not that I objected or was hard to persuade. I am delighted to serve in this capacity, to be associated in this way with a research organization I consider to be outstanding in so many ways. But, I think I finally got the message. An honorary chairman should be seen and not heard--or at least. should not be heard very long. So I will be brief in my remarks.

I do insist, however, in saying some things about the Southern Utilization Research and Development Division which have been on my mind for a good long while. First--and most important--is the very sincere tribute I wish to pay to Hap Fisher and the scientists, engineers, technicians, assistants--in fact, that whole fine group of people who really are the laboratory. Never have I had the opportunity to work with a more cooperative group, and I know that many of you in this audience would say the same thing. This wonderful spirit of cooperation is truly the cornerstone upon which the Southern Division has built its enviable reputation.

It has taken more than a willingness to work with others, however, to bring the laboratory to its position of leadership in cotton research. It has taken a high level of scientific competence, and the kind of dedication that finds some staff members working long hours after closing time and others at their desks and benches on Saturdays and Sundays. And it has taken patience, the patience necessary to put up with the pressures, red-tape, and frustrations which are, at least in some degree, a part of any government operation.

This laboratory has to operate in a kind of never-never land between university science on the one hand and industrial research and development on the other. Make no mistake; this isn't easy. The laboratory is expected to produce positive, tangible, and even marketable results in order to justify its existence and earn the appropriations that keep it alive. Yet at the same time the laboratory is expected not to preempt the functions of private industry, and this is especially important, because in the long run no development is going to be commercially successful unless the legitimate incentives of private industry are preserved and protected.

As I said, this isn't an easy position to occupy. The laboratory is subject at the same time to charges of transgressing industrial preserves and of failing to bring its developments to full commercialization. Yet to an increasing extent as the organization has matured it has found a solid, productive place in this limbo. Fundamental studies--the type of research on which private industry can best build its own commercial applications--have increased at the Southern Division from about 20 percent of their total research a few years ago to around 35 percent today, and, I understand, is to be further increased to around 50 percent.

This very meeting is evidence of another constructive move of the Southern Division to get its findings to industry at the earliest possible stage. I think the presence of this fine audience fully demonstrates that this is proving highly successful.

There is no question that the Southern Division is of tremendous importance to the raw cotton industry. It is essential. No matter what we do to make cotton competitive in price, no matter how much we promote it, we cannot move it into consumption unless it provides those qualities and characteristics which consumers demand. The Southern Division has made many important contributions to our knowledge of how to improve cotton's mill processing characteristics, and how to capitalize on its great potentials in chemical reactivity. I am sure these contributions will accelerate. Therefore, it gives me great satisfaction to be associated in a small way with this important meeting here today.

OPENING REMARKS

by
J. Lindsay Dexter
Pepperell Manufacturing Company
Boston, Massachusetts

The papers which you are to hear during this Fifth Cotton Utilization Research Conference represent a selection of the outstanding projects of the current program of the Southern Utilization Research and Development Division and are only a small part of the great effort being expended in the cause of cotton utilization research by a group of dedicated men and women. Put into simple language, it means that they have been laboring mightily on how to get people to use more cotton. Translating all this into the jargon of the Great Society, their goal is to escalate the use of cotton.

The problem facing cotton today can be divided into two categories: Economic and Technological--and the two are inextricably intertwined. Economically, the goal is to raise, harvest, process, and finish cotton at a price that will compete not only with foreign cotton growths but with synthetics at home and abroad. Technologically, the goal is to produce a cotton with the best possible fiber characteristics, to grow, to harvest, and to process it, with a minimum of damage, into fabrics and other products with attributes that are equal or superior to any blended or all-synthetic product. The economic goal can be attained only through research on growing, harvesting, processing, and finishing methods which will produce cheaper yet superior products--in other words, through technology.

Only through a fundamental understanding of the structure and properties of the cotton fiber and of the mechanics involved in its chemical and physical behavior can a sound program be formulated for investigating the optimum mechanical forces and chemical reactions needed for processing cotton fibers and fiber assemblies. Exploratory chemical and physical investigation can provide the building blocks for the processes and products of the future.

As a result of their splendid program of basic research, both physical and chemical, the Southern Division has made great contributions towards the development of new and better cotton products, by pioneering in such fields as easy-care finishes, fire retardant and rot resistant finishes, all-cotton stretch yarns and fabrics. In addition, the basic research now under way into the effect of electrostatic and aerodynamic forces on cotton fibers could very possibly result in some significant developments in cotton processing machinery and methods in the near future. Through their study of the interrelation between cotton fiber properties and processing efficiency, they have been of great help to the mills in determining the optimum machine adjustments to handle various fiber conditions. Judging from the projects proposed for the future at the Southern Division, there is much to look forward to.

Great emphasis is being placed on the importance of easy-care and permanent press finishes, and rightly so, as our competitors, the synthetics, have made great strides in this direction. However, one thing that must not be lost sight of in the mad rush to match the easy-care properties of the synthetics is that cotton has certain peculiar attributes which give it comfort characteristics unequalled by either blended or 100 percent synthetic products. In our efforts to improve cotton easy-care properties, let us be careful not to sacrifice any of its comfort characteristics.

The textile industry in general and the cotton industry in particular are on the verge of a great new era. Mills with carefully controlled conditions of temperature and humidity will be the rule. Working conditions will be greatly improved through filtering of dust and reduction of noise. Machinery speeds and efficiency will be increased and mechanical-handling equipment will speed the progress of the material through the mill. Finishing plants will be designed to treat, wash, and dry fabrics at a rapid rate without chemical damage and without distortion or strain to the cloth being processed.

Marketing of cotton will undergo a radical change due to the development of automatic instruments for rapid measurement of all the

fiber properties necessary for determining the value of cotton to the spinner. Cleaning and blending will be done by ginning factories under controlled conditions so as not to damage the fibers' spinning value. Cotton will be bought and sold on fiber specifications and blends will be tailormade by the gin factory to suit the mill customers' requirements. The mill, in turn, will be able to verify the quality of the fiber being delivered by statistical sampling and testing with high speed automatic instruments.

When these things come about, and they will before too long, it will be due in no small part to the solid foundation of basic knowledge supplied to the industry by institutions such as the Southern Utilization Research and Development Division and its dedicated staff. But with all these wonderful new developments will come an added challenge to keep on expanding the use of cotton through utilization research.

THE EFFECT OF FABRIC STRUCTURE ON THE PROPERTIES OF COTTON STRETCH FABRICS

[Summary]
by
G. F. Ruppenicker, Jr. and J. J. Brown
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by G. F. Ruppenicker, Jr.)

The effect of fabric structure on the properties of fabrics woven from crosslinked cotton stretch yarns was investigated. Both filling-stretch and 2-way stretch fabrics were woven using various weaves and fabric constructions. Fabrics with from about 10 to 50 percent usable stretch were produced. In most cases, constructions with less than the normal number of ends and picks per inch were used in order to allow the stretch yarns room to relax and contract during washing and desizing operations. A change in the number of ends or picks per inch had considerable effect on the amount of fabric stretch. Sateen and other weaves with long floats produced fabrics with the most stretch. Minimum stretch was obtained in the filling direction by using a warp rib weave and in the warp direction by using an oxford weave. In general, flat abrasion resistance increased as the amount of stretch was increased.

To obtain a variety of physical and esthetic properties, methods of finishing and aftertreating the stretch fabrics were evaluated. The relaxed stretch fabrics were treated with from 2 to 8 percent solids solutions of the same resin used to treat the yarns, resulting in an increase in the resin add-on of from approximately 1 to 4 percent. The aftertreated fabrics had less stretch but much better recovery than the untreated stretch fabrics. Dimensional stability and wrinkle recovery after repeated home launderings were significantly improved by a resin add-on of 3 to 4 percent. Fabric strength decreased from 5 to 30 percent compared to untreated stretch fabric. The flat abrasion resistance of the aftertreated stretch fabrics was slightly less than that of the untreated stretch fabric, but equal to that of untreated, nonstretch fabric of comparable structure.

Fabrics woven from the stretch-type cotton yarns were also aftertreated using a delayed curing procedure to impart permanent creases. The stretch fabrics were padded with the resin solution and dried smooth on a tenter frame. They were then made into trouser cuffs, pressed, and cured. The treated trouser cuffs exhibited excellent wrinkle resistance, crease retention, and wearing qualities after 30 home launderings.

DISCUSSION

Question: I am interested in knowing more about the washing and drying conditions used on the trouser legs subjected to 30 home launderings.

G. F. Ruppenicker, Jr.: We used a standard home laundry procedure for "wash-and-wear" items as outlined in the AATCC Technical Manual, consisting of a 4-pound load and high-water level. The trouser cuffs were tumble-dried for 40 minutes between each wash cycle.

Question: Has any work been done on coating these fabrics?

G. F. Ruppenicker, Jr.: These fabrics have been designed primarily for apparel uses, whereas coated fabrics would have industrial uses.

Question: Did you evaluate the dimensional stability of the aftertreated fabrics?

G. F. Ruppenicker, Jr.: Yes, filling-stretch fabrics aftertreated with a 6 to 8 percent resin solution shrunk less than 1 percent warpwise and less than 2 percent fillingwise after 15 home launderings, which should be adequate for most end uses.

Question: Did you run any tests comparing the properties of these fabrics with slack mercerized resin-treated fabrics?

G. F. Ruppenicker, Jr.: Generally, more "easy" stretch can be obtained by this method of producing stretch fabrics.

Question: Were the permanently creased stretch fabrics compared with similar permanently creased conventional fabrics after repeated home launderings?

G. F. Ruppenicker, Jr.: Yes, both conventional and stretch permanently creased fabrics were laundered 30 times together. The stretch fabrics showed no abnormal wear, whereas some of the conventional fabrics developed holes in the cuffs.

THE SRRL BALE-OPENER-BLENDER--A PROGRESS REPORT

[Summary]
by
J. I. Kotter and R. A. Rusca
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by R. A. Rusca)

In the textile industry the practice of blending cotton is almost as old as the industry itself. In the earliest days of spinning, individual mills obtained their cotton from a small geographical area and the need for blending was negligible. Later, blending was primarily for the purpose of controlling the color and cost of raw materials rather than for technological reasons. This situation has changed radically. Improvements in machinery and increasing labor costs and competition have resulted in the industry converting to fewer prespinning processes, fewer doublings, much higher drafts and machine speeds, and larger packages--all placing added stresses on cotton fibers. It is now vital that an adequate number of bales be blended in textile mills to assure the production of quality products at low costs.

In the past few years new equipment for blending called "bale digesters" or "bale pluckers" has been introduced. Bale pluckers are essentially automatic hopper-feeders. These advantages and disadvantages are too well known to require discussion here.

In 1959, the Southern Utilization Research and Development Division conducted a survey of blending practices, costs, and needs. Based on this survey, research was started in January 1960 on the development of an improved method for blending cotton that would meet five objectives: (1) Thoroughly blend at least 20 bales in any desired proportion, without the use of additional equipment to achieve the actual blending; (2) production rate of about 1,000 lbs./hr., adjustable over a wide range, and maintained constant at any desired rate regardless of bale size, density, or weight; (3) deliver tufts of cotton weighing not less than 0.1 g. or more than 0.5 g. into either a suction duct or a conveyer belt; (4) process the entire bale without waste or need for discarding the last part of the bale; and (5) physical size of the system not to exceed that of the hopper-feeders required to give an equivalent production.

The principle of the machine is simple. Layers from a single bale are laid end to end on a conveyer belt. On top of these layers are placed other layers in staggered relationship until a 20-layered sandwich is formed. The conveyer moves the continuous bat into the blending machine against two horizontal processing rolls with relatively large teeth set in a spiral pattern. The rolls traverse the face of the bale in 2 seconds, subjecting the face of the bale to about 47,000 teeth per minute. The small tufts of cotton fibers removed from the bale are discharged through a chute onto a conveying belt or into an air duct.

Evaluations of a pilot scale blender indicate that a commercial machine would blend up to 1,000 lbs./hr., with a tuft weight of 0.150 to 0.450 g. depending upon production, and without fiber damage or neps. The cotton is blended in exactly the same proportion as the sandwich bale was prepared. Mill trials of a full size machine are scheduled later in the year.

DISCUSSION

Question: How is the degree of blending assessed, by running conventional spinning tests, or is a colorimeter used?

R. A. Rusca: Because our textile laboratory is equipped with one hopper-feeder only, it was not possible to compare the Bale-Opener-Blender with standard blending procedures. Blending was determined by measurements of fineness and fiber length distribution. The colorimeter would be useful in evaluating the efficiency of blending cottons of widely different colors, spots, stains, and so forth.

Question: How are the original bales separated, how is the sandwich bale formed?

R. A. Rusca: Layers of cotton are manually removed from the bales that are to be blended. These layers are laid end to end on a conveyer belt. Successive layers are placed on top of the first layers in staggered relationship until a 20-layer sandwich batt is formed on the conveyer. This procedure eliminates the problem of choking and nonuniform tufts resulting from the end of the bale.

SHORT FIBER REMOVAL BY ELECTROSTATIC FORCES

[Summary]

by
J. J. Lafranca, Jr., M. Mayer, Jr., and H. W. Weller, Jr.
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by J. J. Lafranca, Jr.)

Research has been conducted to develop means for fractionating lint cotton into long and short fiber length groups. Short fibers are defined as those shorter than 3/8-inch in length. Removal of these short fibers from lint cotton improves strength, uniformity, and appearance of cotton yarns, and permits the more efficient processing of all cottons, including difficult-to-spin cottons.

Electrostatic force was chosen as the medium of separation of fibers by length as a result of preliminary research. This study of the basic mechanisms of fiber motion under the influence of electrostatic fields revealed conditions under which short fibers act differently than long fibers. The problem of separating short from long fibers was then reduced to the design of a force field and associated components with sufficient sensitivity and control to operate on a continuous basis.

In operation, the input cotton is first drafted to the density of card web. Then it is fed between a feed plate and a high speed bristle brush where the web is individualized and directed into an electrostatic field. Once in this field the fibers are aligned along the electrostatic lines of force. The longer fibers are attracted to one portion of the field at a greater rate than the shorter ones, thus affecting separation. Nonconducting doffing devices remove the long fibers directly from this region, and the short fibers are conveyed to a point where they are accumulated on felt wipers.

Several promising experimental machines utilizing specially designed electrostatic fields to fractionate fibers have been developed. Two of the machines are 1/5 scale models capable of operating directly on an input of card web. These can be readily widened to 40 inches for use with the conventional textile card. Typical results from laboratory tests on the machines are given below:

Production rate	Short fiber input	Content output	Short fiber reduction	Long fiber removal[1/]
gm./hr.	%	%	%	%
27	8.6	5.1	40.8	7.0
69	8.6	6.3	27.0	2.8
114	8.6	6.9	19.8	3.4
173	8.4	6.6	21.4	3.6
213	9.0	7.7	15.1	1.5

1/ Percent of long fibers in the input removed with the short fiber fraction.

CRITICAL LOOK AT COTTON SPINNING DRAFT DISTRIBUTION

[Summary]
by
G. L. Louis and L. A. Fiori
Cotton Mechanical Laboratory
Southern Utilization Research and Development Division

(Presented by G. L. Louis)

The effect of various combinations of draft-zone variables, e.g., total draft, back draft, tensor setting, and roll setting, on yarn properties and end breakage is becoming increasingly more important, especially since the trend in the textile industry is toward the use of higher and higher total drafts. Heretofore, spinners have been using the manufacturer's recommended draft distribution. However, there are indications that better distribution of drafts may be needed to obtain minimum end breakage at some selected level of yarn strength, and vice versa.

This paper reports limited investigations conducted on the effect of draft distribution on spinning performance and yarn properties using three drafting systems; viz., the double apron Casablanca system (referred to as System A), the Duo Roth modified double apron system (System B), and the Shaw apron-roll combination system (System C). Two 1-1/16-inch cottons (an Acala 4-42 and a Delta cotton) and a 1-inch cotton of unknown growth history were used. Four separate experiments were conducted in this investigation. The SRRL 720 Spindle-Hour Test and the SRRL Accelerated End Breakage Test methods were used for the end breakage evaluations.

Experiment No. 1 covered the relationship among back draft, total draft, tensor setting, end breakage and yarn properties on drafting System A using a Delta cotton. Three tensor settings, 4 mm., 5 mm., and 7 mm.; three total nominal drafts, 30, 40, and 50; and three back drafts, 1.3, 2.1, and 2.7 were used resulting in 27 combinations. It was found that end breakage increased curvilinearly as back draft and total draft increased. Also, end breakage difference between the 30 and 40 total drafts was less when spun with tensor setting of 4 mm. than with 5 mm. and 7 mm. Moreover, a tremendous difference in end breakage occurred (over 280 times) when extreme draft-zone combinations were used, viz., 30 total draft--1.3 back draft--4 mm. tensor setting, and 40 total draft--2.7 back draft--7 mm. tensor setting. Yarn strength increased gradually as front draft increased for total drafts of 30 and 40 while no changes occurred when front draft increased on the 50 total draft. Maximum strength was obtained with back draft of 1.3 in combination with a total draft of 30. Statistical analysis of yarn data showed that an increase in total draft significantly affected yarn strength and elongation-at-break. Increases in back draft affected yarn uniformity very significantly while varying total draft and tensor setting affected yarn uniformity to a lesser extent.

Experiment No. 2 covered a comparison of the three drafting systems, A, B, and C using 1-1/16-inch Acala 4-42 and a 1-inch cotton. The relationship between back draft and end breakage of all three drafting systems studied can be described by a type of curve that has a minimum point and the two ends of the curve turning upward. This relationship indicates that regardless of drafting system used, there exists a back draft which yields minimum end breakage and beyond or below which end breakage will increase. Also, the back draft which yields minimum end breakage does not always produce yarn having maximum strength. In other words, the back draft which gave best levels of end breakage and yarn strength varies from system to system.

Experiment No. 3 covered an investigation of the effect of spacings between front roll and the aprons on drafting System A using an Acala 4-42 cotton. The spacings between the nip of the front rolls and the tip of the apron used were approximately 0.61, 0.67, and 0.73-inch. End breakage decreased curvilinearly while

yarn strength, elongation and uniformity increased curvilinearly as the distance between apron and front roll were decreased. The decrease in end breakage was much sharper than the improvement in yarn properties indicating that the apron to front roll setting was more critical to end breakage than to yarn properties.

Experiment No. 4 covered an investigation of the effect of roving twist on spinning performance when using an Acala 4-42 cotton on System A. The three tensor settings and back drafts used in Experiment No. 1 along with total drafts of 15, 20, 30a, and 40 were used to spin the Acala 4-42 cotton into 30/1 and 40/1 yarns with a 4.00 Twist Multiplier (TM) from 1.0 and 2.0 hank rovings each having 1.04 and 1.22 TM's. This investigation indicated that small differences on roving twists do not affect end breakage in any combinations of back draft and tensor setting except when the 1.3 back draft and 4 mm. tensor setting are used in spinning the "hardtwist" roving. However, the same lots spun with back draft of 2.10 and 4 mm. tensor setting presented no difficulty.

DISCUSSION

Question: What hank roving did you use in Experiment No. 1? If different sizes of roving were used, did the roving twists remain the same?

G. L. Louis: The rovings used in Experiment No. 1 were 1.03, 0.76, and 0.61 hanks having Twist Multipliers of 1.02, 1.06, and 1.07, respectively. The range of these TM's is rather small as compared with the Twist Multiplier level of about 1.60 used in industry.

Question: If you were to plot roving twist against end breakage in spinning, what relationship would you get? Do you have any control experiment where hank rovings were constant?

G. L. Louis: No attempt was made to investigate the effect of roving twist on end breakage because the differences of these twists were so small. The rovings used in Experiment No. 1 were meant to have common Twist Multiplier. With cotton Twist Multiplier but different hank roving the twist per inch of the three rovings used would have to be different because T. P. I. = TM $\sqrt{\text{Hank Roving}}$.

Question: How do you explain the behavior of the back draft-end breakage curve? Why does the difference in back draft affect end breakage?

G. L. Louis: The reasoning that higher back draft has less fiber control may be used to explain the portion of the curve where back draft increases from 1.3 to 2.7. But the same reasoning does not hold when back draft drops from 1.3 to 1.2 and below; therefore, we really do not have the proper explanation to the question. Perhaps the spinning manufacturers will find an answer.

RECENT ADVANCES IN WOOL RESEARCH

[Summary]
by
H. P. Lundgren
Wool and Mohair Laboratory
Western Utilization Research and Development Division
Albany, California

Although it is among the oldest textile fibers, wool, strictly speaking, was not designed by Nature to be used by man. Wool is a hair manufactured by the sheep and for the sheep. It is made in the follicles, the small "test tubes" in the skin of the sheep. Even though this process is strictly the sheep's business, it is nevertheless instructive to consider the follicle as a manufacturing assembly line. Several kinds of cells are lined up along the walls of the follicle. These cells are arranged to perform their functions in sequence--an arrangement much like that of a modern assembly line. Each kind of cell does its part toward making the wool fiber; and with a 24 hour-a-day production schedule the end product, wool, comes out quality controlled and magnificently adapted to protect the sheep against the rigors of sheep's environment--but we are not sheep.

Modern man needs wool that will withstand the rigors of scouring, carbonizing, bleaching, crabbing, backwashing, dyeing, flat setting, automatic machine washing, drying, and--in general--the dirt, grime, and smog of our manufactured environment. To meet such requirements--and this is indeed a big order--it is necessary to find effective and economical ways of modifying those parts of wool's structure that lack proper resistance to damage. To go about such modification systematically requires that a good part of our work be basic in nature.

Basic Studies in Structure and Properties

Anatomically the wool fiber is wonderfully complex and very difficult to copy. Evidence indicates that the characteristic keratin molecules of wool are helically coiled polypeptide chains united in groups of 3 to form protofibrils. As many as 9 protofibrils appear to be arranged in a shell concentrically around 1 or 2 central protofibrils to form the microfibril, the 20 Angstroms-in-diameter fundamental fibrous unit of wool. It is the α-keratin protein of the microfibril that gives the X-ray diffraction patterns from which Astbury 30 years ago first concluded that the characteristic protein molecules of wool are curled up, and that they uncurl when the wool fibers are stretched.

The microfibrils themselves are embedded in a nonfibrous protein matrix packaged neatly into macrofibrils which in turn are packed together to make up the cells of the cortex. The cortex comprises 90 percent of the wool fiber substance. Surrounding the cortex is a laminated cuticle--a skin made up of three and possibly four structurally distinct proteins.

In developing basic knowledge of the fiber stability we use special tools. For example, we have adapted the election spin resonance technique to study mechanical damage of wool. This method is based on the principle that mechanical action that damaged wool (such as in processing) produces unpaired electrons in the fiber. Such unpaired electrons appear when the disulfide bonds that crosslink wool are severed.

Rapid and precise assessment of mechanical properties of wool is a particular research need and essential to improvement of wool products and processes. A single fiber stress-strain curve digital computer has been built at the Western Regional Research Laboratory to greatly increase the speed and precision of measurement of wool's stress-strain parameters.

One of the unique properties of wool is its ability to be flexed repeatedly over sharp bends without breaking. This ability may reflect its unique internal structure. On the other hand the outside skin may play a part. The presence of an appropriate skin is known to affect the mechanical properties of diverse materials.

Steel ball bearings, for example, are now made with an outer steel skin in a state of compression. This skin helps distribute external stresses so that they do not concentrate on flaws inevitable in the usual steel surface, and the new ball bearing is more durable.

It is possible to show by means of photoelastic birefringence that thin films properly deposited on the surface of a birefringent plastic redistribute applied stress over a notch purposely put in the surface, and permit repeated flexings under conditions where the untreated plastic easily breaks at the notch.

Significantly thin coatings of 6-10 nylon polymer grafted to wool by our interfacial polymerization method significantly increase the ability of wool to be flexed repeatedly. In other words the number of flexings to break are increased. Abrasion resistance is also significantly increased by the film coating.

By means of our interfacial polymerization method various kinds of polymer films are being anchored in the surface layers of wool. This will enable systematic study of surface modification on fiber mechanics.

Phase Boundary Limiting Crosslinking

A new technique has been found in our laboratory for forming durable finishes on wool through crosslinking of reactive polymers. The wool is impregnated with an organic solution of a reactive polymer and subsequently treated with an aqueous solution of the crosslinking reagent to form a polymeric finish which is insoluble in all types of solvents, and therefore durable through launderings and dry cleanings. By this technique--which we call phase boundary-limited crosslinking--wools have been treated, for example, with polyamides and polyurethanes for shrink-resistance, and with polysiloxanes and fluoropolymers for resistance to water and oil. Many commercially available, reactive polymers are suitable for this application, and a wide range of properties can be built into the treated fabrics.

This new treatment can use polymers that our WURLAN method cannot, and so widens considerably the range of polymeric finishes possible for wool. Many polymers and crosslinking agents which can be used are commercially available and potentially cheap. We anticipate pilot plant studies on the most promising of these to help translate our findings into commercial use.

New Fluoropolymers

The present commercial fluorine-containing compounds that are applied to wool fabrics to make them water and oil resistant are relatively expensive. Some very promising inexpensive fluorine-containing compounds were synthesized in our laboratory and we are now screening them to see if they can be used to finish wool fabrics. The new fluoropolymers are based on derivatives of hexafluoroacetone. Since the price of this compound is predicted to be less than $1 a pound, treatment of fabric with the new compounds should be considerably less expensive than similar treatments now used. Preliminary evaluations indicate that hexafluoroacetone-based polymers are durable to both laundering and dry cleaning. After several launderings, treated fabrics retain excellent oil and water repellency. We hope to translate the more promising results into commercial use.

Control of Yellowing

The yellow color of many wools has become an important problem in recent years with the increasing demand for white and pastel fabrics. Buyers pay premiums for exceptionally white wools. Many wools are more or less yellow and some wools are rather deeply colored. The yellow colors in general are not removed by washing and are even difficult to remove by bleaching. Bleaching, furthermore, damages the fiber strength and other properties of wool.

Our greatest progress toward practical control of yellowing has been in a treatment which prevents yellowing of shorn wool that is damp or wet. Often when the wool is sheared, the fleece is damp (or even wet)--and always it is dirty. Damp and dirty, it is baled and, perhaps kept for some time. Under these conditions the wool yellows and eventually weakens. This undesirable yellowing is caused by a combination of chemical and microbial action. It can be prevented or minimized if the wool is dusted with paraformaldehyde just prior to

baling. About 1 percent of the aldehyde by weight is sufficient to keep the temperature from rising within the bale and almost eliminates the yellowing. Wool also yellows on long exposure to light. Research is under way to find ways of minimizing such damage. Significant progress has been made toward understanding the mechanism of photochemical degradation.

Wool also yellows by heat--for example scorching on ironing. Such damage is promoted by perspiration residues in wool. Recent studies in our laboratory demonstrate that such residues can assist in splitting sulfur from its combination in the keratin protein crosslinks. The free sulfur in wool favors diverse reactions with peptides and proteins, especially oxidation; the products are yellow to brown.

Resistance to Hydrolysis

Hydrolytic evaluation of wool, which is necessary for an understanding of acid and alkaline degradation mechanisms and as background for developing more stable wools, is under experimental investigation and theoretical consideration in our laboratory. An electronic computer program was written to help in applying the theory. Experimental data on differences in the rates of hydrolysis of wool, and of other keratins for comparison, theoretically account for differences in sulfur content as directly relating to crosslinking. It is possible from the information obtained to estimate the proportion of disulfide bonds effectively crosslinking different chains. Knowing this proportion is helpful in our program to improve the stability of wool. In parallel studies of structure, our organic chemists are coming up with modifications having greater resistance to alkalies and acids.

Commercial Use of WURLAN-Treated Wool Top

Before concluding, I should like to demonstrate the newest end-product of WURLAN treatment. This is an all-wool 2-piece ladies knit suit. The yarn for this suit was made from WURLAN-treated wool top. Like fabrics treated by the WURLAN process, the knit goods (which are difficult to treat directly) have machine washability, enhanced resistance to pilling, enhanced resistance to abrasion; colors are level and fast. The top can be dyed after treatment. The wool does not lose weight by the treatment. The handle is good. Moisture uptake, and hence comfort in wear, is unchanged. And the effects of the treatment are durable.

Finally, I should like to emphasize that, although we have made what we feel is significant progress, there is much left to do. Possibilities are open for new fiber modifications. It is conceivable that we can develop a multipurpose finish that gives wool in one treatment more desired effects than presently possible, such as permanent resistance to insects, mildew, and wrinkling, along with dimensional stability and neatness retention. Possible are new kinds of yarns and fabrics with new and desired textures. We feel that scientifically designed, superior, man-modified wools are beginning to answer the threat to wool's markets by the manmade fibers. America is spending close to $30 billion annually for apparel. The markets for textile fibers are growing along with our population, which is expected to reach 225 million by 1975. The share that the natural fibers will have in tomorrow's textile markets will, as always, be determined by the qualities these fibers offer in relation to their price.

CHANGES IN FINE STRUCTURE OF COTTON FIBERS WITH GROWTH

[Summary]
by
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The cellulose in the cotton fiber while still growing in the boll is in a porous, amorphous state or is crystalline from the beginning. The dense, crystalline structure which is found in cotton after harvesting could have resulted from a solidification when the fiber, naturally very moist while growing, began to dry out for the first time as the boll opened. This crystalline structure is a useful property of cotton fiber, contributing to the strength of yarns and fabrics; but it also tends to produce brittleness in fabrics treated with some of the modern textile finishing agents. A less dense--more accessible--cellulose structure would be advantageous when resins and crosslinking chemicals are to be applied to produce wash-wear or drip-dry fabrics. A more accessible cellulose can be obtained by pretreatment with the proper swelling agents; but it would be of interest if the fresh, undried cotton fibers were found to be naturally highly accessible and could be treated directly with crosslinking chemicals.

It seemed worthwhile, therefore, to re-examine the important question of when the crystalline structure develops in the fiber. This paper describes various tests which were applied to fibers removed from fresh, moist cotton bolls and preserved in different ways chosen to maintain the moist condition until testing. The results of these tests indicated rather definitely that the undried fibers actually are already partly crystalline. The dense, highly crystalline structure of the mature fiber develops gradually as the plant deposits the cellulose in the cell wall. An additional definite but small increase in crystallinity also occurs when the fiber first dries out. The large change upon initial drying that earlier workers had shown was not found in the present study. The results of the present study are based on entirely different techniques from those used by earlier investigators. Also, instruments and techniques have been much refined since then and this can contribute to the more certain conclusions of the present study.

With regard to the possibility of using moist, fresh fibers as a highly accessible material for applying textile treatments, our results suggest the futility of such an approach since the cotton fiber apparently is already of fairly high crystallinity even before growth has been completed.

DISCUSSION

Question: You mentioned a possible mechanism by which highly crystalline cellulose could be laid down outside the cell membrane. From my concept of the cotton fiber I cannot visualize how this can take place. What evidence is there?

M. L. Nelson: The bacterial cellulose is synthesized outside the bacterial cell; an energy-rich glucose derivative is the cellulose precursor from which an enzyme system synthesizes cellulose microfibrils, apparently by adding glucose units one at a time to the ends of the microfibrils.

Comment: But that is for bacteria, not cotton. There is no liquid outside of the cell wall.

Comment : In the morphology of the fiber, the foot extension of the fiber is below the seed coat cells. Building blocks for cellulose (that is, glucose) are transported through the seed-coat to the fiber. Glucose is produced in the leaf and is transported through the vascular system to the seed and thence to the growing fiber.

C. M. Conrad: Analyses of the growing fibers show that as much as 50 percent of the sap is sugar, so there is plenty of sugar available for synthesis into cellulose.

Question: If free energy requirements cause chain-folding in regenerated cellulose, why isn't there chain-folding in cotton cellulose?

M. L. Nelson: There are two or three current theories of chain-folding in native cellulose; Manley in Montreal, and Ellefsen and Sippel in Europe have proposed different models. The mechanism of synthesis I mentioned would be equally acceptable for folded chains as for straight chains.

Question: But what about the theories of cellulose being synthesized within the protoplasm and being extruded through the cell membrane?

M. L. Nelson: Perhaps Miss Rollins would like to comment on this.

M. Rollins: Very interesting new concepts about production of cellulose are being advanced by Porter and Ledbetter at Harvard; by Muhlethaler and Frey-Wyssling at Zurich; and by Wardrop in Tasmania on the microtubule theory. This is very new and will bear watching.

COTTON FIBER STRUCTURE AND PHYSICAL PROPERTIES ALTERED BY ENVIRONMENT

[Summary]
by
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Southern Utilization Research and Development Division
and
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(Presented by J. N. Grant)

Fibers from Deltapine 14 and Rex varieties grown under field and under four controlled environments were examined for differences in wall structure and for relationships of wall structures to their physical properties. Environmental conditions were chosen to secure altered structure caused by (a) varying light and varying temperature; (b) continuous light and constant temperature; (c) varying light and constant temperature; and (d) continuous light and constant temperature. Except for the diurnal rings found only in fibers grown under conditions which had a varying temperature, no large structural differences were found in fibers from the different environments. The number of days between flowering and boll opening was a minimum when light was continuous and the temperature was constant. The period increased about 20 percent (8 days) when both the light and the temperature were varied. The development of the secondary wall and the formation of reversals followed a pattern generally found in field grown cottons.

Fibers with differences in diurnal ring structures were swollen with a methanol-water mixture and embedded in methacrylate. The rupture patterns for the secondary wall were similar for cottons of different structures and no obvious relation could be detected between the position of rupture and the diurnal ring. The ruptures were, in general, parallel to the plane of the primary wall. At low magnification of the electron microscope, segments of secondary wall separated by the polymerization of the methacrylate appeared to form concentric circles. Under higher magnification, the secondary wall was found to have ruptured into segments of cellulose of rather uniform thickness but varying in dimensions parallel to the primary wall. The comparable rupture pattern for cottons of the different growth conditions, even though differences in diurnal ring structures were found, is evidence that the diurnal ring structures are layers of cellulose. Sufficient differences in density could make them visible but no evidence was found that discontinuities were present which caused preferential cleavage at the diurnal ring structure.

Environmental conditions were found to affect length but their influences on fineness and maturity were small. Greater differences were found between physical properties of field-grown and controlled-growth cottons than among the cottons of different controlled growths. Cellulose orientations (X-ray) of the field-grown cottons were appreciably different from those of the controlled environments. A significant relationship was found between cellulose orientation and bundle tenacity at zero gage. Equally as good as relationship was found for cellulose orientation and bundle elongation. The cottons grown under conditions which formed no diurnal rings at comparable cellulose orientation had higher elongation at break and lower tenacity then those with the rings. A low tenacity in field grown cottons is common when conditions such as high temperature and moisture promote rapid plant growth. The differences in percentages of crystalline cellulose (X-ray) for cottons of the various growths are insufficient to conclude that tenacity and elongation differences are caused by the amounts of crystalline cellulose. An environment which causes rapid plant growth could

produce a more open secondary wall structure. Consistent with the assumption that the more open structure is responsible for the higher elongation is the greater decrease in bundle tenacity and elongation of the resin-treated fibers without the diurnal rings than those with the rings. Apparently the resin bonds are more effective between fibrils of the open structure since no diurnal rings are present. This leads to the conclusion that crosslinks are formed between fibrils throughout the secondary wall rather than at the diurnal rings as previously assumed to be the locations that the bonds are formed.

DISCUSSION

Question: I assume the fibers were from opened bolls and that cuprammonium was used to assess ring structure. From previous research I have concluded that growth rings were differences in cellulose structure and that differences were obviously more dependent upon temperature than on light. The boll would more likely respond to temperature than to light. I am concerned about growth rings and our interpretations. Will properties such as chain length be examined?

J. N. Grant: The report is preliminary and made from the properties we have measured. The work is by no means complete either in production of fibers of different structures or in analyses of structures on fibers we have grown.

Question: Was the reversal frequency determined?

J. N. Grant: The frequency was determined for the Deltapine cotton fibers grown under three environments: Field grown, variable temperature and light, and constant temperature and light. For each of these the frequency was found to be 29 reversals per cm.

Question: Have the fibers been fractionated and the longitudinal structure examined?

J. N. Grant: The internal structure from longitudinal rupture of fibers has not been examined. From the cross sections, the rupture pattern, and the physical properties, one would not expect a large structural difference. It is possible that this difference is too insignificant to be detectible.

Question: Was a load pull-down made on any of the fibers before drying?

J. N. Grant: Studies on the fibers before the boll opened have been very limited, but efforts will be made to determine the properties of the undried fibers and their relation to wall development.

THE ELECTRON MICROSCOPY OF SOME CHEMICALLY MODIFIED COTTONS

[Summary]

by
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(Presented by M. L. Rollins)

The objective of microscopical investigations of textiles is to study changes in fiber structure which may accompany changes in physical properties induced by various chemical finishing treatments with a view to explaining the behavior of textile products. This paper describes microscopical evaluation of chemically modified cottons, including the effects of chemical degradation, slack-and tension-mercerization, the breaking and reforming of crosslinks, and graft polymerization.

When cotton fibers are beaten apart in water in a laboratory blender, untreated fibers yield sheets of fibrils in which microfibrils 100-200 Angstrom units wide and several microns long are arranged in parallel fashion. Cottons containing intermolecular crosslinks form short, jagged fragments consisting of several layers of fibrils; the appearance of fibrillar shearing and fracture is related to the type and extent of crosslinking. Fragments from substituted cotton (cellulose esters or ethers) have an amorphous appearance; the greater the degree of substitution, the more amorphous material and the fewer the discrete fibrils obtained. Fragments from cotton mercerized in the slack condition show a certain amount of fibril disturbance, and evidence of fibrillar crimping. In cotton degradation, the distinctive pattern of fragments from acid hydrolysis is that of short, needle-shaped "micelles"; in contrast, oxidation produces fragments which are much less discrete, and which appear swollen. Fragments exposed to various swelling agents on the specimen grid revealed differences in fibrillar behavior; after swelling in concentrated ethylenediamine, 70 percent ethylamine, 5 percent sodium hydroxide, or in saturated calcium hydroxide, the rinsed and dried specimens differed little from the untreated control, but crimping of fibrils was a striking feature of the behavior of fragments in 18-percent potassium hydroxide and 18-percent sodium hydroxide. Two successive treatments with 18-percent sodium hydroxide resulted in fusion of the fibrillar elements in the sheet. Full strength "Triton B" brought about dissolution of fibrils and the formation of short particles, although a 50-percent aqueous solution had no visible effect.

Application of the technique for separation of the cell wall into layers has demonstrated that differences exist between products of slack and of tension mercerization. The same technique for expanding the internal structure has shown that interlamellar and intralamellar crosslinking are usually correlated with the chemical history and physical behavior of the fiber. In the wash-wear field, it is of interest to produce fabrics in which it would be possible to break and reform the crosslinks locally. In exploratory microscopical investigations, it has been possible to show the effect of such treatments. Sections of fibers crosslinked by a reactive dye showed no layer separation by the expansion technique; but when the dye was reduced, the resulting fibers separated like untreated cotton. In recoupling, following diazotization of the reduced cotton, it was found possible to restore the interlayer bonding to a situation similar to that originally induced by the sulfone dye as crosslinking agent.

It has been proposed that formation of rubbery polymers within the fiber could improve abrasion resistance and elasticity. When cotton is irradiated with a high-energy source (gamma rays), free radicals are believed to be created within the cellulose molecules, permitting graft polymerization of such polymers as styrene and acrylonitrile to cotton. Electron miscroscope observations have determined the location and uniformity of such grafts within the fiber.

While more precise methodology exists for chemical analysis of reacted cellulose, electron microscopy offers the best approach for location of structural alteration within the fiber, which bears on the physical behavior of yarns and fabrics.

DISCUSSION

Question: Have you crosslinked these grafted samples and examined properties such as strength and abrasion?

M. L. Rollins: I would like to refer this question to Mr. Arthur.

J. C. Arthur, Jr.: Crosslinking of these grafted samples is being studied but results are not yet conclusive.

PHOTOCHEMICAL DEGRADATION OF COTTON [1/]

[Summary]
by
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Photochemical decomposition of cotton, cotton mercerized with and without the application of tension, partially acetylated cotton and partially cyanoethylated cotton during exposure to sunlight and to radiations from carbon-arc lamp of a Fade-Ometer, under controlled conditions of exposure--viz. presence or absence of oxygen, humidity in the atmosphere, selected wavelength bands in the ultraviolet, and visible region of the solar spectrum, etc. --has been studied. Extent of degradation has been determined from changes in breaking strength, cuprammonium fluidity and formation of carbonyl groups, total carboxyl groups, and uronic acid carboxyls.

With respect to solar exposure, the different fiber substances show considerable differences in the extent of mechanical as well as chemical damage and the degrading action of sunlight is not only confined to short wavelength radiations but radiations from longer wavelength regions also bring about significant decomposition. Humidity of the atmosphere has marked influence on degradation and the effect is different for the different fiber substances examined. Chemically modified cottons behave differently during photosensitization in presence of phototending vat dyes and the extent of damage produced depends on conditions of humidity and sequence of operations involved during chemical modification. Kinetics of the photochemical decomposition reaction reveal that photodegradation proceeds mainly by random chain scission.

1/ This work forms part of the research being carried out at the University under Public Law-480; this grant was issued by the Agricultural Research Service, United States Department of Agriculture.

ENERGY TRANSFER IN FIBROUS COTTON CELLULOSE AS RELATED TO ITS THERMAL, PHOTOCHEMICAL, AND RADIOCHEMICAL RESISTANCES

[Summary]
by
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(Presented by J. C. Arthur, Jr.)

The energy absorbed by fibrous cotton cellulose, either from incident high energy radiation, from photochemical radiation, or from thermal radiation, results in similar effects on the cellulose molecule--oxidation, activation of excited sites, evolution of gases including hydrogen, carbon dioxide, and carbon monoxide, and degradation of the molecule particularly through depolymerization. These chemical changes cause losses in breaking strength of the cotton and in the case of weathering may accelerate the degradation of the cotton. For photochemical energies, a few electron volts, energy absorption is dependent on molecular structure and, therefore, is chemically specific. For high energy, several million electron volts, energy absorption is not dependent on molecular structure and, therefore, is not chemically specific. However, intramolecular, and possibly intermolecular, transfers of both absorbed photochemical and high energies within the cellulosic molecule result, as indicated, in localization of energy in groups or bonds of the molecule to initiate similar changes in chemical and physical properties of the fibers. Processes for increasing the resistances of cotton to degradation by directing intramolecular, and possibly intermolecular, energy transfer to and localization in chemically substituted groups, from which the energy can be dissipated, are discussed.

Energy transfer processes in matter in the solid state are generally considered to be dependent on the mechanism of energy loss by the incident radiation to the chemical molecule, the initial random nonlocalized deposition, and subsequent dissipation, of the energy within the molecule, and the rapid localization of the energy within the molecule. The localization of the energy within the molecule results in chemical and physical changes in the molecule. An early example of radiation protection was that when benzene was added to a hydrocarbon and the mixture irradiated, a lower yield of gaseous products was received than when the hydrocarbon was irradiated separately. The radiation protective effects of aromatic groups have since been the subject of many investigations, and a number of explanations has been advanced to account for their protective effects. Generally, it has been concluded that either energy transfer and/or charge transfer to the aromatic group occurred from which the energy was dissipated without causing chemical decomposition. It has also been suggested that the mechanism of the radiation protective effect of benzene, considering the π-electron system, was one of selective energy absorption rather than energy transfer. Further, it was suggested that through the mechanism of selective energy absorption, energy of secondary electrons produced by γ-radiation was absorbed by the aromatic group.

When carbohydrates in the solid state are irradiated by high energy radiation, the localization of the absorbed energy within the molecule and resultant chemical effects are dependent on the physical structure of the carbohydrate, on certain complex formation of other molecules with the carbohydrate, and on the substitution of chemical groups on the molecule. In the case of solutions of chemically substituted carbohydrates of low molecular weight, substitution of aromatic groups on the molecule influences both the excitation and the fluorescent spectra of the solutions. When the substituted chemical group on the glycoside was benzoyl, the excitation and fluorescent spectra of solutions of these products had principal bands with approximately equal wavelengths. The fluorescent spectra exhibited slightly longer wavelengths than the excitation spectra, indicating

that most of the energy absorbed by the solution on excitation has reemitted during fluorescence. The effects of aromatic groups on the radiation protection of solid glycosides are summarized in table 1. Benzoyl groups were more effective than phenyl, methyl, and acetyl groups in protecting the glycosides from radiation degradation. In other work it has been shown that benzyl groups are more effective in their protection than phenyl groups but less than benzoyl groups.

Table 1.--Effect of aromatic groups on the radiation protection of solid glycosides[1/]

Glycoside	Melting point (°C.)	Product	G[2/]
Methyl α-D-gluco-pyranoside	167	Glucose Acid	2.33 1.45
Tetra-O-acetyl methyl α-D-glucopyranoside	101	Glucose Acid	2.33 7.9
Methyl α-D-mannopyranoside	193	Mannose	2.0
Phenyl β-D-glucopyranoside	174	Glucose Phenol Acid	0.8 .8 .5
Tetra-O-benzoyl phenyl β-D-glucopyranoside	160	Glucose Phenol	[3/] .05
Tetra-O-benzoyl methyl α-D-glucopyranoside	104	Glucose	[4/]
Hepta-O-acetyl phenyl β-maltoside	155	Phenol Cleavage -glycosidic -disaccharide	.05 .05 .10

1/ From Phillips, G. O., Blouin, F. A., and Arthur, J. C., Jr., Radiation Research 23: 527 (1964) and Nature 202: 1328 (1964).

2/ Molecular actions per 100 electron volts of energy absorbed.

3/ At dosages as high as 6.16×10^{21} ev/g, glucose was not measurable.

4/ At dosages as high as 4.55×10^{21} ev/g, glucose was not measurable.

These investigations were extended to the protection of fibrous cotton cellulose from degradation on exposure to high energy radiation and to weathering, by preparing benzoylated cottons. The radiation resistance and the weather resistance of benzoylated cotton yarns are summarized in tables 2 and 3.

Table 2. --Radiation resistance of benzoylated cotton yarns[1,2,3]

Degree of substitution	Unirradiated		Irradiated	
	Breaking strength (lbs.)	Elongation-at-break (%)	Breaking strength (lbs.)	Elongation-at-break (%)
	Purified cotton yarn			
0.0	10.5	15.9	2.7	9.6
	Restretched treated yarn			
.8	9.8	15.0	8.0	13.0
1.1	12.6	11.2	7.9	7.9
1.3	12.8	12.4	10.3	11.4
	Slack treated yarn			
1.3	5.2	23.7	4.2	21.9
1.5	6.5	34.2	5.5	39.7

1/ From Arthur, J. C., Jr., Mares, T., and George, M., Textile Res. J., In review.
2/ Instron properties.
3/ Cobalt-60 gamma radiation dosage: 25 megarads.

Table 3. --Weather resistance of benzoylated cotton yarns[1,2]

Weathering, weeks	Breaking strength of yarns, lbs.			
	Cotton control	Degree of substitution		
		0.6	1.1	1.5
	Yarns restretched before drying			
0	11.7	11.0	11.1	8.7
10	5.8	9.4	11.2	7.6
26	3.1	8.6	9.9	9.6
	Yarns dried slack			
0	11.7	-	-	3.9
13	2.6	-	-	3.2
26	1.5	-	-	3.6

1/ Adapted from Arthur, J. C., Jr., Mares, T., and George, M., Textile Res. J., In review.
2/ Instron properties.

It can be concluded that the presence of the benzoyl groups affected the absorption of incident energy and/or the localization of the energy within the molecule. In the case of weathering, the protective effect of the benzoyl group could be due to the selective absorption of the incident light energy and reemission of the energy as light or heat. In the case of exposure to high energy radiation, the secondary electrons produced on interaction of the high energy with the cellulose molecule could be directly absorbed by the benzoyl groups, and

the energy also be reemitted. In both cases, the incident energy, even if absorbed, would be transferred in such a way as to minimize localization at a chemical bond which could cause degradation and loss in strength of the fiber.

Results from preliminary experiments on thermal initiation of the degradation of cotton cellulose, as evidenced by changes in free radical spectra, indicate that the intramolecular transfer and localization of absorbed energy from incident thermal energy can also be directed.

DISCUSSION

Question: Does it make any difference if the glycoside is the alpha or beta anomer?

J. C. Arthur, Jr.: No, it does not.

Question: Does the break in the curve of strength retained vs. degree of substitution have any significance?

J. C. Arthur, Jr.: Yes, the break occurs at a D.S. of approximately 1.1, at which by statistical calculations every cellobiose unit would have at least one benzoyl substituent.

Question: Does the fact that the substituent is attached through an ether or an ester linkage have any influence on the protective effect?

J. C. Arthur, Jr.: Yes, we have found the benzoyl group (ester linkage) is much more effective than the benzyl group (ether linkage) in protecting the carbohydrate molecule.

IODIDE ION CATALYSIS IN CROSSLINKING COTTON WITH ORGANIC CHLORIDES

[Summary]

by
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Southern Utilization Research and Development Division

(Presented by R. H. Wade)

The literature reports very little about catalysis of cellulose reactions with organic halides and, in most instances, dissolution of the fiber is involved. Reaction conditions that retain the fiber per se impose limitations on catalyst and reagents due to the multiphase system and diffusion rates. The diffusion problem may be reduced by placing the catalyst in contact with the reactive sites of the fiber. The problem still remains for any subsequent agent that must diffuse to the reaction site. At the reaction site the "catalyst" must activate the reaction and regenerate itself.

The alkali metal nitrite, thiosulfate, borate, and iodide were screened as part of the caustic treatment in the benzylation process. Since the alkali metal iodide was exceptionally effective, initial work was confined to the iodides using benzyl chloride as a model. Addition of small amounts of the iodide reduced the substitution below the control without iodide, but increased concentration of the iodide resulted in an increased degree of substitution to about twice the control value. The iodide salt level was maintained at 20 percent and the caustic concentration varied. The results show that mercerizing strength is needed to obtain high substitution. The effective moiety was shown to be the iodide ion. The strength of the benzylated yarn was affected only at high temperatures and long reaction times. The strength of the benzylated yarn was not affected by the presence of iodide.

The general effectiveness of iodide catalysis was shown for monochloromethyl compounds such as substituted benzyl chlorides and chloro methyl naphthalene as well as for alpha-omega dichloro compounds such as dichloropropene, dichlorobutene, and para-xylylene chloride.

Results of solubility tests with organic cellulose solvents and calculations based on elemental analysis and weight gain indicate that crosslinking takes place with the polyfunctional chloro compounds.

DISCUSSION

Question: Are you able to assess the relative contribution of cellulose swelling by sodium iodide to the catalytic action of this salt?

R. H. Wade: We do not think that the additional swelling contributes much to the catalytic action under mercerization conditions. The effect of sodium iodide concentration on the diffusion rate of benzyl chloride in cellulose is not known, but this may affect somewhat the rate of reaction.

Question: Is there any indication that the positions of cellulose substitution are any different with and without the catalyst (iodide ion) present?

R. H. Wade: We do not believe the sites are different, but we do not know. Samples will be submitted to Miss Rollins' group for examination by electron microscopy.

Question: What is the effect of structure of the etherifying agent on its reaction rate with cellulose?

R. H. Wade: The data show wide differences in etherification rate with different organic chlorides. The rate appears to increase with decreasing size and increasing electronegativity of the organic radical to which the halogen is attached. Allylic double or triple bonds also greatly increase the reactivity toward cellulose. Thus the rate of diffusion of the etherifying agent into the fiber is only partly rate controlling.

PREPARATION AND PROPERTIES OF VINYLATED COTTON (VINYL ETHER OF CELLULOSE) [1/]

[Summary]
by
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(Presented by M. E. Chiddix)

The vinylation of simple alcohols with acetylene and other reactants to form vinyl ethers is well known. Although the vinylation of cellulose has been investigated by Shostekovski and other workers, the products reported were characterized by excessive degradation.

The study of the vinylation of cotton which I shall report had the following objectives: 1) To determine whether cotton in fiber form could be vinylated and retain most of its strength and other properties, and 2) to study the reaction of cotton fibers with acetylene. Because of the successful preparation of vinylated cotton slivers and cloth, further objectives were to improve the vinylation process and to make a preliminary measurement of physical and chemical properties of vinylated cotton.

As shown in figure 1, four different methods of vinylation were examined. First, the reaction of cotton cellulose with ethylene dibromide and sodium hydroxide was found to form the bromoethyl ether and the vinyl ether. The final product contained both substituents with a D. S. vinyl ether value between 0. 08 and 0. 26 and a D. S. for bromoethyl ether between 0. 06 and 0. 12. The second method was that of transvinylation using a low molecular weight vinyl ether and a mercury catalyst. Some vinyl ether of cellulose was produced in this way up to a D. S. of 0. 07. However, metallic mercury was deposited in the cotton fibers during the process and was quite difficult to remove. The third method was based on a disclosure of Reppe that vinyl chloride and caustic could be used to vinylate alcohols. This was tried with cotton cellulose, but only a small amount of vinylation resulted. The fourth method, that of vinylation with acetylene using a potassium hydroxide catalyst, proved to be the most effective and provided a process for vinylating cotton over a range of D. S. values up to 0. 70.

Reaction	D. S. Values
1. $ROH + BrCH_2CH_2Br \xrightarrow{NaOH} ROCH_2CH_2Br$	0. 06 - 0. 12
$ROCH_2CH_2Br \xrightarrow{NaOH} ROCH=CH_2$	0. 08 - 0. 26
2. $ROH + C_4H_9OCH=CH_2 \xrightarrow{Hg(OAc)_2} ROCH=CH_2 + C_4H_9OH$	0. 06 - 0. 07
3. $ROH + CH_2=CHCl \xrightarrow{NaOH} ROCH=CH_2$	0. 03 - 0. 06
4. $ROH + HC\equiv CH \xrightarrow{KOH} ROCH=CH_2$	0. 05 - 0. 70

Figure 1. --Vinylation methods.

1/ A report of work done under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act. The contract was supervised by the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

The vinylations with acetylene were carried out in a 1-liter stirred autoclave. Cotton slivers impregnated with the catalyst were suspended in an acetylene solvent, and the mixture was stirred and heated in the autoclave under acetylene and nitrogen pressure. At the end of the reaction, the slivers were filtered, washed thoroughly with THF and water to remove the alkaline catalyst, and vacuum dried.

The slivers were analyzed for vinyl ether both iodimetrically and by hydrolysis to acetaldehyde. Although the hydrolysis method would measure acetal linkages as well as vinyl ether, it was in good agreement with the iodimetric method which is unaffected by the presence of acetal. This is evidence that very little cross-linking by acetal formation has occurred. Examination of the infrared absorption spectrum also provided confirmation of the vinyl ether substitution. A medium intensity band characteristic of vinyl ether was observed at 830 cm^{-1}. It was absent in the control cotton.

In table 1 a comparison of the catalysts examined for the vinylation of cotton slivers with acetylene is shown. Although Reppe had reported the use of cadmium and zinc acetates as catalysts for the vinylation of alcohols, the results obtained with cotton slivers were variable and the values for vinyl ether substitution were in the range of 0 to 0.10. Choline hydroxide, a quaternary ammonium hydroxide, showed some catalytic activity and was tried because of its known swelling action on cotton fibers. Although D.S. values from 0.04 to 0.07 were achieved with cotton slivers, results with cotton cloth were not as good using this catalyst. Sodium hydroxide proved to be much inferior to potassium hydroxide as a vinylation catalyst. In addition, the sodium hydroxide caused much more degradation of the cotton under vinylation conditions.

Table 1.--Vinylation with acetylene comparison of catalysts (6 hr. reactions, cotton slivers)

Catalyst	Conc. (mole, pct.)	Temp. (°C.)	D.S. vinyl ether
$Cd(OAc)_2$	14	120	0.10
Choline hydroxide	100	120	.04
Choline hydroxide	100	140	.07
NaOH	100	140	.05
KOH	100	140	.28

In table 2 the effect of catalyst concentration is shown. The amount of potassium hydroxide on the cotton slivers is expressed as a percent of the weight of the dry cotton slivers. The vinylation reaction was run 6 hours at 120° C. or 248° F. The rate of vinylation was rather low at 17 and 26 weight percent potassium hydroxide based on the cotton slivers. Good rates were achieved at 35 percent without much degradation of the cotton as shown by the 92-percent product recovery. Product recovery is the weight of the dry vinylated cotton expressed as a percent of the weight of the dry cotton used. Although higher vinylation rates were achieved at 52 percent potassium hydroxide, much loss of cotton occurred due to degradation. In each case the weight of residual water based on the weight of the starting cotton was kept the same.

Table 2.--Effect of concentration of KOH catalyst (cotton slivers, 6 hrs., 120°C.)

KOH conc. wt. (pct.1/)	Water wt. (pct.1/)	Product recovery (pct.)	D.S. vinyl ether
17	15	89	0.08
26	17	94	.10
35	15	92	.30
52	15	76	.66

1/ Based on weight of cotton used.

Both the residual moisture in the catalyst impregnated cotton and the acetylene solvent proved to have a significant effect on the vinylation rate as shown in figure 2. With either toluene or dibutyl acetal as the solvent, it was found that the rate of reaction drops as the moisture content of the cotton is increased from 20 to 100 percent of the weight of the cotton. The highest rates of vinylation were achieved at 20 percent moisture and below. Consistently higher rates were obtained in dibutyl acetal as compared to toluene. However, the vinylated cotton was darker, and in the case of cotton cloth more shrinkage and puckering of the cloth was observed when using dibutyl acetal. A number of other solvents were tried including THF, N-methylpyrrolidone, dimethylsulfoxide, pyridine, dimethyl formamide, dimethyl acetamide, and dioxane. Some of these gave good rates of

reaction but caused excessive degradation of the cotton and with others very poor vinylation rates

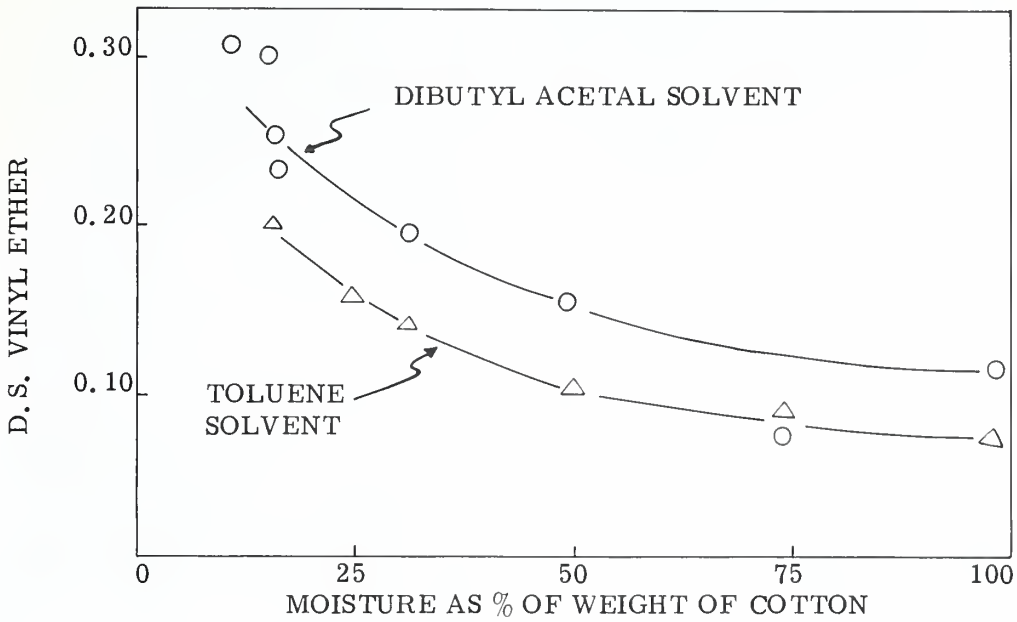

Figure 2. --Effect of moisture on vinylation rate (Temp., 120° C.; Time, 6 hrs.).

were observed. As a result, toluene and dibutyl acetal were used in most of the vinylation work.

The effect of acetylene pressure on the vinylation rate (table 3) was determined in order to obtain a better idea of the type of equipment that might be necessary to handle this vinylation. Low rates were observed at partial pressures of actylene of 20 and 40 lbs. guage, but a sharp increase in rate was obtained on going up to 120 lbs./in.^{2}, and this pressure was used for most of the vinylation work.

Table 3. --Effect of acetylene pressure (vinylation of cotton slivers, 6 hrs., 120°C.)

Acetylene press. (p. s. i. g.)	Nitrogen press. (p. s. i. g.)	D.S. vinyl ether
20	20	0.03
40	40	.07
120	80	.30

Because of the good results obtained with cotton slivers, the study was extended to the vinylation of cotton broadcloth. Strips of cloth 4.75 inches by 36 inches or 72 inches in length were impregnated with aqueous potassium hydroxide, dried quickly in a steam press, and wound around the holder which is pictured in figure 3 giving either 4 or 8 layers of cloth. The cloth was held in place by means of a few loops of thread around the edges. The holder was then placed in the autoclave in place of the regular stirring shaft, and the holder and cloth rotated during the vinylation reaction with the acetylene solvent covering the cloth. Good vinylation of cotton print cloth was obtained in this way.

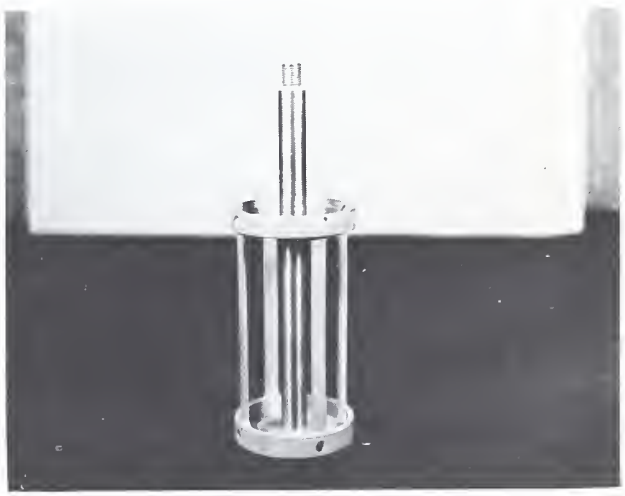

Figure 3. --Cloth Holder

In table 4, an indication of the rate of vinylation of 80-square cotton print cloth at 120° C. is given. The D. S. vinyl ether values increased from 0. 10 to 0. 74 with increasing reaction times from 3 hours to 24 hours. At this reaction temperature very little degradation occurred as measured by weight loss of the print cloth even over a 24-hour reaction period. The vinylation of cloth proceeded more rapidly at 140° C. in other experiments, but more degradation of the cotton was observed and some water soluble or THF soluble vinylated cellulose was produced.

Table 4. --Vinylation of cotton print cloth (toluene, 120°C., 36 percent KOH)

Reaction time (hrs.)	Product recovery (pct.)	D.S. vinyl ether
3	96	0. 10
6	94	. 22
12	98	. 48
24	91	. 74

The cotton slivers and cloth were insoluble in the standard cupriethylenediamine hydroxide solution except for some samples with very low degrees of vinylation. In table 5 we have an indication of the solubility and the D. P. calculated for a few of the soluble samples. On the right hand side it can be seen that only three samples at rather low D. S. values were soluble. These three had been vinylated in the N-methylpyrrolidone solvent. None of the cotton slivers vinylated in dibutyl acetal or in toluene were soluble in the reagent. From the intrinsic viscosities, degrees of polymerization were estimated for the 3 soluble samples. In comparison with the value of 3000 given in the literature for untreated cotton, it is apparent that considerable degradation has occurred during vinylation in N-methylpyrrolidone. The insolubility of the vinylated cotton slivers produced in toluene or dibutyl acetal indicates some crosslinking of the cotton during the vinylation reaction. However, this amount of crosslinking was not sufficient to have any effect on the crease recovery angle of the vinylated cotton print cloth.

Examination of the vinylated cotton by means of X-ray diffraction gave a good indication of the extent to which the crystalline regions of the cotton fibers were affected. Figure 4A shows a photograph of the X-ray diffraction pattern of unvinylated cotton slivers and figure 4B the pattern taken from a sample that had been vinylated to a D. S. value of 0. 14. The loss of the outer circle of light and the bright lines of figure 4A as compared with figure 4B indicates almost complete alteration or loss of crystallinity and preferred orientation. It is thus apparent that the catalyst and acetylene have completely penetrated the crystalline regions of the cotton slivers. The same results were obtained when examining cotton slivers vinylated to higher D. S. values as well vinylated print cloth. At low D. S. values of 0. 05, the X-ray diffraction pattern was altered slightly but did not show the change illustrated in figure 4. Hence, at these low degrees of vinylation very little alteration of the crystallinity was produced.

Table 5. --Solubility and D. P. of vinylated cotton

Vinylation solvent	Temp. (°C.)	Intrinsic viscosity	D. P.	D. S. vinyl	Solubility
Untreated cotton	--	18. 11	(3000)	--	Soluble
NMP 1/	110	3. 09	586	0. 02	Soluble
NMP 1/	120	1. 40	266	. 01	Soluble
NMP 1/	140	0. 52	97	. 08	Soluble
DBA 2/	110	--	--	. 07	Insoluble
Toluene	120	--	--	. 27	Insoluble

1/ N-Methyl-2-pyrrolidone
2/ Di-n-butyl acetal

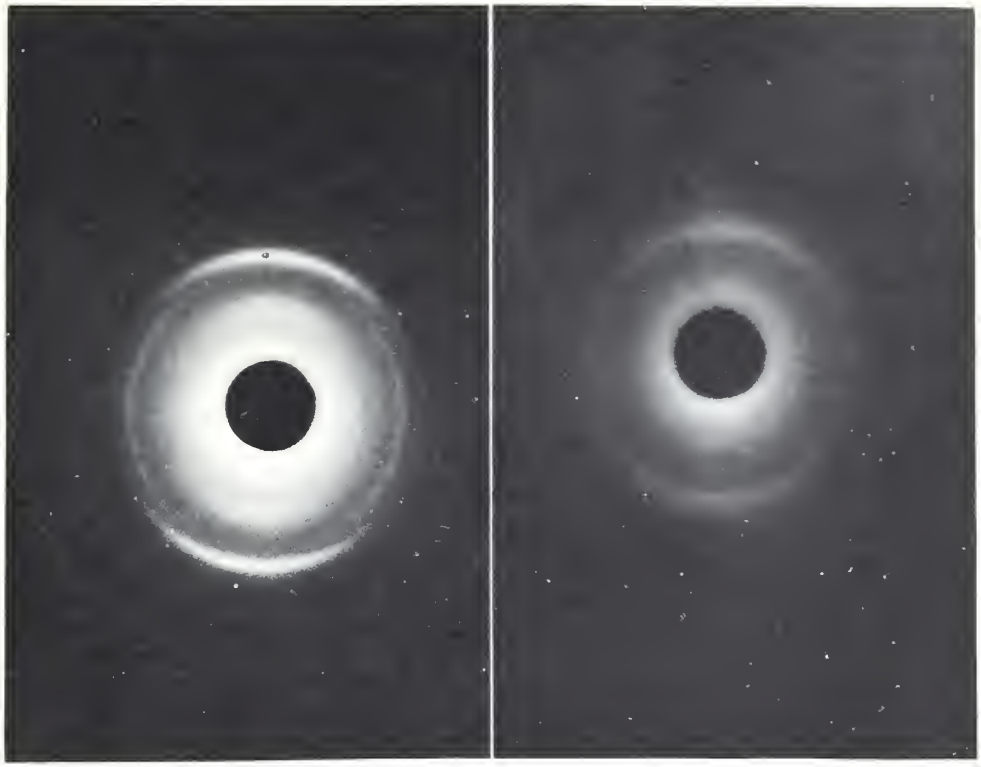

Figure 4. --Flat plate X-ray diffraction patterns

A. Unvinylated cotton B. Vinylated cotton (D. S. =0. 14)

The breaking strength of the vinylated cotton print cloth was examined with an Instron tensile tester with the results shown in table 6. The samples range between 55 and 93 percent of the breaking strength of the control cloth. Since considerable shrinkage had occurred, the breaking strength was calculated on a basis of pounds per strand. In all cases, the percent elongation at break increased due to the treatment with 26 percent aqueous potassium hydroxide catalyst. No correlation was observed between breaking strength and D. S. value. It was demonstrated, however, that vinylated cotton print cloth with good breaking strengths can be obtained although no process has been developed to control this factor.

Table 6. --Breaking load for vinylated cloth
(80-square cotton print cloth)

Sample	Breaking load percent of control	Percent elongation	Strands per inch	D. S. vinyl ether
Control	100	8	80	--
1	85	28	101	0. 30
2	88	28	101	. 16
3	93	37	104	. 22
4	55	53	94	. 17

In order to establish that the treatment with the alkaline catalyst alone was not responsible for the loss of tensile strength, a study was made of cloth treated with the catalyst and then washed free of the catalyst. In table 7, it can be seen that there is some effect due to the catalyst but only over extended periods of time.

Table 7. --Breaking load--alkali-treated cloth

Sample	Hours contact	Breaking load percent of control	Percent elongation	Average strands (inch)
Control cloth 136 x 68	--	100	10	144
1	16	98	30	144
2	66	86	29	146
3	16 1/	93	36	147

1/ Contract time: 16 hours wet + 24 hours with 25 percent water.

Essentially no loss in tensile strength was observed after 16 hours at room temperature although the elongation increased. In the vinylation procedure, cloth was soaked only 1 hour in the aqueous potassium hydroxide before drying.

In order to determine the effect of the catalyst under vinylation conditions, a sample of cloth was impregnated with catalyst and heated in the autoclave with nitrogen in place of acetylene. After completing the regular vinylation procedure, the observed breaking strength was 105 percent of that of the control cloth. The elongation was 95 percent vs. 10 percent for the control. Most of the loss of tensile strength is apparently due to the action of the acetylene. In view of the X-ray results, this may be due primarily to the loss in crystallinity.

In order to determine the reactivity of the vinyl ether groups of the vinylated cotton, a crosslinking reaction was attempted using two different acidic catalysts. Dilute aqueous solutions of the acidic catalysts were padded on vinylated cotton cloth with a D.S. value of 0.21. The cloth was air dried for 5 minutes, and then pressed dry on a flat bed press. The cloth was then heated for 3.3 minutes in the oven at 300° F. In table 8 the crease recovery angles and the breaking strengths of the resulting cloth are given. Improved crease recovery angles were obtained with the amine hydrochloride catalyst without loss of breaking strength, and higher crease recovery angles with the $MgCl_2$ catalyst although with considerable sacrifice of breaking strength.

There was not sufficient time during the contract to find the optimum conditions for crosslinking the vinylated cotton or for exploring other possible reactions.

Table 8. --Crosslinking and wrinkle recovery of fabrics (ATTCC test No. 66-1959T)

Crosslinking catalyst	Crease recovery angle (degree)		Breaking strength (lbs.)
	Dry	Wet	
Control (none)	108	181	17
Catalyst AC (Amine. HCl)	211	234	17
$MgCl_2$	312	252	5

In conclusion, it was demonstrated that vinylated cotton slivers and cloth could be prepared with good retention of fiber strength. X-ray diffraction patterns indicated penetration of the crystalline regions during vinylation with acetylene. Good crease recovery values were obtained by heating samples of vinylated cotton print cloth with acidic catalysts.

We wish to acknowledge the assistance of C. H. Mack and C. R. Stahl of our laboratory for physical testing and analysis, respectively. We would like to express to the management of

the Southern Utilization Research and Development Laboratory our appreciation for the funds and the opportunity to do this contract research.

DISCUSSION

Question: Are the vinylated cottons thermoplastic; that is, on ironing can a permanent crease be placed in the fabric?

M. E. Chiddix: No attempt was made to determine whether a permanent crease could be imparted to the vinylated cotton by ironing.

Question: Was there any indication that polymerization had occurred in the fiber?

M. E. Chiddix: Although some small degree of crosslinking occurred which made the vinylated cotton fibers insoluble, it is more likely that this was due to acetal formation by reaction of vinyl ether groups with hydroxyl groups on adjacent cellulose chains instead of polymerization. Vinyl ether monomers do not ordinarily undergo peroxide catalyzed homopolymerization.

WASH-WEAR FINISHING AGENTS FOR COTTON BASED ON HYDROXYALKYL CARBAMATES

[Summary]
by
R. M. H. Kullman, J. G. Frick, R. M. Reinhardt, and J. D. Reid
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by R. M. H. Kullman)

New carbamate finishing agents for cotton, methylol derivatives of hydroxyalkyl carbamates, have been developed that produce wrinkle-resistant and wash-wear fabric with little adverse effect on lightfastness of dyed cottons.

Hydroxyethyl carbamate, $HOCH_2CH_2OCNH_2$, is a typical and probably the most useful compound for making these agents.

Preparations of the methylol derivatives are essentially the same as for alkyl carbamates previously described by workers of this Laboratory. However, more care is required to maintain the necessary alkaline conditions during reaction with formaldehyde.

Comparisons were made with other finishing agents on effects of shade changes of 4 main classes of dyes due to crosslinking and effect on lightfastness. Formaldehyde condensates of hydroxyalkyl carbamates were superior to those of alkyl carbamates and ethyleneurea and comparable to a urea formaldehyde type finish.

Other properties that were examined on fabrics finished with the new agent included wrinkle resistance, resistance to chlorine damage, and durability. Careful curing is required to obtain full chlorine resistance, however, durability to chlorine resistance is less than for alkyl carbamates. Other properties, including durability of wrinkle resistance, were comparable to alkyl carbamates.

In tests to show applicability of the new agents for deferred cure processes, a 66-day delay before curing had no adverse effect on properties of finished fabrics. Reduction of formaldehyde to carbamate ratio to reduce formaldehyde odor on uncured fabric had little effect on fabric properties.

DISCUSSION

Question: Can you give a range of curing conditions explored with dimethylol hydroxyethyl carbamate?

R. M. H. Kullman: Curing conditions for samples reported here were 3 minutes 160°C.; it is important to obtain the cure temperature or less resistance to chlorine damage will result.

Question: Were samples washed before Fade-Ometer testing of colorfastness? Residual catalyst may affect colorfastness.

R. M. H. Kullman: All the samples were washed after the carbamate finishing step and presumably contained no residual catalyst during Fade-Ometer testing.

Question: Can you comment on what advantages might be expected from the use of dimethylol hydroxyethyl carbamate instead of a dimethylol dihydroxyethyleneurea-type agent in deferred curing?

R. M. H. Kullman: The chlorine resistance and durability of the dimethylol hydroxyethyl carbamate deferred cure finish appears to be much better than that of currently used deferred cure finishes.

Question: How long does it take to reach full chlorine resistance when the lower molar ratios of formaldehyde are used in deferred curing?

R. M. H. Kullman: Less than 30 days. No curing was done between 0 and 30 days storage.

STRETCH LACE WITH ATTRACTIVE ESTHETIC PROPERTIES

[Summary]
by
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W. A. Reeves, and M. J. Hoffman
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by H. M. Robinson)

A three-dimensional lace with a reembroidered textured look produced by a simple inexpensive process--this is the dream of the lace industry come true. The Southern Utilization Research and Development Division, U.S. Department of Agriculture, in New Orleans has produced just that from an ordinary Leavers-woven inexpensive cotton lace. The treatment gives to a flat cotton lace a raised third dimension in parts of the design and in addition stretch properties. The resulting lace is richer, handsomer, and much more expensive looking than the original.

The treatment used is simple and most inexpensive in operation. Since the process can be applied to finished lace, difficulties encountered in making lace from stretch yarns are avoided. Manufacturers are thus able to manufacture the lace in their usual manner on their present machines, and even in the patterns they are already using.

The process, slack mercerization, is already in use by a number of commercial finishers for the production of all-cotton stretch fabrics and has now been applied to lace with startling results in dimensional pattern changes.

The slack mercerization treatment to produce stretch properties is highly versatile; amount and direction of stretch can be "tailored" to the requirements of the use to which the fabric or lace is to be put. The stretch is durable and any small losses in resilience occurring during repeated wearings are promptly recovered in laundering. Stretch fabrics are also improved by resin treatment, which gives them wash-wear properties.

The slack mercerization-resin treatment can be applied in regular finishing equipment. The cost is low as generally estimated by manufacturers now applying it to standard fabrics. There is some loss in dimensions, roughly equal to the amount of stretch produced, but this will be easily swallowed in the greatly improved appearance which increases the value of the finished lace.

Samples of laces purchased from retail stores were used and these varied from lightweight, plain all-over laces, to clunys and corded laces. All samples took on stretch properties and showed improvement in richness of appearance. This latter property was especially outstanding in the least expensive samples.

The enriched lace is praticularly attractive used in clothing and the sample dresses and coats used to demonstrate its attractions have aroused much interest. Aside from the added beauty of the treated lace, the "comfort stretch" added by the process assures better fit at less expense, and greater durability. We feel that this is the first cotton fabric offered that can easily hold its own as a formal fabric in the High Fashion World.

DISCUSSION

Question: Do you think the lace could be preshrunk before it is put on the market so that the 10 percent the washed sample looses would be eliminated?

H. M. Robinson: Yes.

Question: Have you measured the treated lace for the amount of increase in thickness or height of design?

H. M. Robinson: No, we haven't. This would vary, of course, depending on the kind of design.

I did notice, however, that a pile of 25 samples of treated lace was 25 to 30 percent higher than a pile of the same lace untreated; and this was true of two quite different laces.

Question: You say ordinary finishing machinery could be used but lace comes off the machines 18 to 20 feet wide and finishing machinery is not that wide.

H. M. Robinson: The wide lace is cut for handling as it comes off the machine, so it would be a size that could be manipulated.

FOURTH SESSION: J. W. Weaver, Chairman

REVIEW OF METHODS FOR PRODUCING DURABLY CREASED AND PRESSED COTTON FABRICS AND GARMENTS

[Summary]

by
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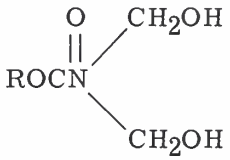
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

One of the most important textile developments in recent years is the availability of durably creased cotton wash-wear garments. A review of methods for producing durably creased cotton reveals the prominent role played by scientists of the Southern Utilization Research and Development Division in research leading to the commercialization of these products. Besides methods presently used by the industry, this review also cites techniques for imparting durable creases which are of theoretical interest and which may provide the basis for future successful commercial processes.

Methods for durably creasing cotton may be categorized in five broad classes: deferred curing, two-stage finishing, recuring, garment treatment, and thermoplastic creasing. Except for the last-named category, all processes are based upon crosslinking the cellulose molecules which make up the cotton fiber while the garment is in the desired configuration. The crosslinks hold the fiber in the creased position during wear and return them to this position after laundering.

At the present time the most widely used method for producing durable creases is that of deferred curing. Basically, the process entails impregnating yardgoods with a solution containing a crosslinking agent for cellulose and a catalyst, drying the yardgoods under conditions which do not bring about reaction between the finishing agent and cotton, fabricating a garment from the goods, carefully pressing to crease and shape the garment, and then ovencuring to bring about crosslinking of the fabric.

Most commercial deferred cure processing utilizes an imidazoledinone crosslinking agent. Carbamate derivatives of the general formula:



(in which R represents an alkyl or hydroxyalkyl group) have been developed at the Southern Division which appear to be well suited for this type of finishing. They are inexpensive; fabric impregnated with the agent but not cured is stable to relatively prolonged storage without appreciable loss of reaction effectiveness; the properties of the fabric are not adversely affected by omission of an afterwash; and the finish imparted has excellent stability to laundering, even under somewhat abusive conditions.

Research activity on durable creasing is at a high level. All phases of the materials and methods of durable creasing are being investigated by various segments of the textile and related industries. New finishing methods, new finishing agents and catalysts, new curing equipment, new fabric structures, and new garment fabrication techniques are being sought. Also, improvement of presently established materials and methods are being studied.

Poorer wear resistance than is desirable has caused a shift by the apparel industry from the use of all-cotton fabrics for durably creased garments to the utilization of fabrics of cotton/synthetic fiber blends. A considerable research effort is being aimed at upgrading the wear resistance of durably creased cotton to realize gains in cotton consumption through this textile development.

The combination of durable creases and wrinkle resistance has taken cotton out of the realm of minimum-care or touch-up apparel into that of truly wash-and-wear garments.

RESEARCH TO IMPROVE ABRASION RESISTANCE OF CROSSLINKED COTTON

[Summary]

by
W. A. Reeves, A. S. Cooper, Jr., and R. J. Harper
Cotton Finishes Laboratory
Southern Utilization Research and Development Division

(Presented by W. A. Reeves)

The early durably creased and pressed men's trousers made of 100 percent cotton exhibited poor resistance to abrasion, especially to edge abrasion such as at creases and at the bottom of trouser cuffs. Much less abrasion occurs with light weight fabrics used in ladies' sports clothes than in 8- to 9-ounce fabrics used in the production of durably creased men's trousers.

Several months ago the Southern Utilization Research and Development Division began to expand its research program to improve abrasion resistance of durably creased and pressed all-cotton goods. We are attempting to develop (a) badly needed basic information, (b) more reliable test procedures, and (c) practical methods for improving the abrasive resistance. The following list gives the general approaches being studied to develop methods for improving the abrasive resistance: (a) Polymer coatings on fibers contained in fabric, (b) blends of cotton and treated cotton, (c) preferential crosslinking within fabric structures, (d) attachment of side groups to the cellulose molecules, (e) fabric structure, (f) slack mercerization, (g) high strength yarn and fabrics, (h) curing conditions, (i) softeners, and (j) combinations of the above.

The primary purpose of this discussion is threefold: (a) To report some differences in the failure of crosslinked cottons as compared to noncrosslinked cotton when abraded, (b) to point out some variables that influence test conditions for measuring abrasion resistance, (c) to point out some fabric and finish modifications that improve the abrasion resistance of crosslinked durably creased fabrics.

Scanning electron micrographs have provided good information of what happens to a crosslinked fiber when abraded to rupture. These micrographs show that the fibers appear to have broken rather sharply. There is little evidence of fibrillation. This is in contrast to what has been observed on noncrosslinked fibers which fibrillate extensively when abraded to rupture. Perhaps the lack of fibrillation in the crosslinked fibers is due to the intermicrofibril and lamella crosslinks.

The abrasion resistance of a fabric is influenced by three factors: (a) Fiber mechanical properties, (b) yarn and fabric structure and, (c) the finish on the fabric. The fiber properties that have the greatest influence on abrasion resistance include strength, both tensile and shear, elongation, elasticity, interfiber friction, and fabric stiffness. Of those properties listed, elongation and elasticity probably exert the greatest influence on abrasion resistance.

There is no known single laboratory test that will enable one to predict the performance of crosslinked or noncrosslinked cottons for various end uses. Perhaps the reason for this is that there are so many variables that influence test results. For example, one can alter the relative abrasion resistance of crosslinked and noncrosslinked cellulosic fabrics by merely increasing or decreasing the load of the abrading force. Thus, if one reduces the abrading force sufficiently a crosslinked cotton may exhibit better abrasion resistance than a noncrosslinked cotton. Another variable in test conditions is the effect of moisture content of a fabric at the time the test is run. Generally speaking for noncrosslinked cotton, the most rapid abrasion occurs with dry fabrics and the rate decreases as fabrics are tested that contain higher and higher moisture contents. This general relationship is also true for crosslinked cotton. However, there is a very significant difference between noncrosslinked and crosslinked cottons. Wet noncrosslinked cotton fabrics in an accelerator for 4 minutes at 2,500 r.p.m. lost 70 percent as much weight as a dry fabric under the same conditions, whereas

crosslinked wet cotton fabric lost only about 35 percent of the weight lost by a crosslinked fabric abraded dry.

Fabrics containing plied yarns generally exhibit significantly better abrasion resistance than fabric containing singles yarn. With other fabric properties being essentially equivalent, fabrics containing 40/2 yarn in warp and fill exhibited about a threefold improvement in flex abrasion over a similar fabric containing 40/1 yarn.

The abrasion resistance as measured by several test methods can be improved by increasing the elongation of the fabric. The improvement is obtained when either singles yarn or plied yarns are used in the fabric but the greatest improvement is obtained when plied yarns are used. For example, the flex cycles to rupture were increased nearly fourfold in a fabric made of 40/2 yarn in both filling and warp by increasing the elongation (easy stretch) from 16 to 24 percent by slack mercerization.

Another very excellent way of improving the abrasion resistance of durably creased and pressed cotton fabrics is through the application of tough polymers to the surface of fibers contained in the fabric. A large selection of polymers are effective in bringing about this improvement. Three examples are nylon applied from a solvent, silicones from either solvent or emulsion, and urethanes applied from emulsions. These can double the wear life of cotton at creased areas as measured by washing and tumbling dry durably creased trouser cuffs. The improvement in abrasion resistance obtained with these polymers can be added to the improvement obtained by slack mercerization. Thus, crosslinked durably creased slack mercerized fabrics containing one of the previously listed polymers have exhibited as much as a threefold improvement over a durably creased cuff which did not contain these polymers and was made from nonslack mercerized fabric.

DISCUSSION

Question: What types of nylon and urethane polymers were used in the experiments that exhibited the greatest abrasion resistance? Could you give the structure or trade names?

W. A. Reeves: The nylon was a methoxy methylated soluble nylon which could polymerize further or react with cotton. The urethane was of the blocked type. It would also polymerize further when it was heated on the cotton.

Question: The structure of the fabric is important to the abrasion problem. The National Cotton Council has observed that the warp yarn at a crease often disappears completely. Would you comment on this?

W. A. Reeves: This will be brought out by Messrs. Vix and Cheatham in their discussions.

Question: Is there a laboratory abrasion test that predicts the service life of a fabric?

W. A. Reeves: No, not to our knowledge, even laundry tests are lacking. There seems to be a big variation in results obtained in different washers and dryers. It has been reported, however, that the wear life of durably creased trousers may be doubled if worn between launderings.

Question: What catalyst was used for the crosslinking agent when a silicone was present?

W. A. Reeves: $Zn(NO_3)_2$.

EASY-CARE COTTON FABRICS WITH UNCHANGED ABRASION RESISTANCE, BY MEANS OF CROSSLINKED SILICONE FILMS

[Summary]

by
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(Presented by C. M. Welch)

It has been widely accepted that high levels of wrinkle resistance and wash-wear performance are obtainable in cotton fabrics only by covalent crosslinking of the cellulose. The fact that most wash-wear agents yield polymers under the conditions of processing is thought to be largely incidental. In previous studies, the application of polymers or polymer-forming materials failed to impart wash-wear properties whenever cellulose crosslinking did not take place also. In a number of instances the applied polymers were three-dimensional networks, and it therefore seemed that crosslinks in the resin had negligible effect on wash-wear properties, as compared to crosslinks in the cellulose.

The present report describes the discovery of exceptions to these generalizations. In the course of studies on coatings for outdoor fabrics, it was observed that a variety of saturated organic polymers undergo very rapid crosslinking when heated in the presence of an organic peroxide. This process can be made to take place in the bulk state, or equally well in films and coatings of the polymer on fabrics. The polymer and the organic peroxide are simply oven-cured on fabric, after application from aqueous emulsion. The polymer is insolubilized as a film surrounding individual fibers, and cannot be removed by solvent extraction. With a number of polymers, the crosslinked films are much more resilient than are the uncrosslinked films.

When polymer-peroxide-water emulsions are cured on cotton fabrics, the crosslinking is found to take place exclusively in the polymer coating and not in the cellulose. The intrinsic properties of the native cotton cellulose fibers are thus preserved. Among the polymers that can be applied in this way are polyvinyl esters, polyvinyl acetals, polyvinyl ethers, vinyl chloride--acrylic ester copolymers, linear aliphatic polyesters, dimethyl silicones, and methyl hydrogen silicones.

Of particular interest are the crosslinked silicone finishes, since they impart greatly increased wrinkle resistance to cotton in either the wet or dry state, together with actual wash-wear performance. The efficiency with which the silicone is crosslinked increases rapidly in passing through a certain molecular weight range, so that the frequency of crosslinking is readily estimated from variation of efficiency with silicone viscosity. Under varying conditions, crosslink spacings of 300-2000 monomer units were found.

Using a commercial dimethyl silicone of molecular weight 130,000, peroxide curing was found to raise the wet and conditioned wrinkle recovery angles by 100° over the values obtained without peroxide present or without any treatment. Values obtained on 80 X 80 printcloth were in the range of 251-265° (conditioned) and 242-269° (wet). Wash-wear ratings of 3 were observed. Flex abrasion resistance remained at 86-139 percent of that for untreated fabric, while tearing strength retention was 105-107 percent. The stiffness was only 56-73 percent of that for untreated cloth. Silicone add-ons of 8-10 percent were used. The fabrics were considerably water repellent, with spray ratings of 80-90. With silicones of high molecular weight, curing at 160°C. (320°F.) for two minutes in the presence of 2.5 percent benzoyl peroxide was as effective as longer, more severe conditions.

On twills, ginghams, and sateens, wrinkle recovery angles in the range of 264-279° (conditioned) and 245-282° (wet) were observed. Wash-wear ratings of 3.7-4.3 which were noted after treatment were 2.0-2.7 units higher than before treatment. The flex abrasion resistance retained varied considerably with the fabric

weave used, but was often in the range of 100-400 percent for sateen or twill. Tearing strength was unaffected in most cases.

On a highly lustrous broadcloth woven of tension-mercerized yarn, the treatment left the luster largely unchanged, but imparted a wrinkle recovery of 283° (conditioned) and also 283° (wet). The wash-wear rating was 4.0 after treatment and 1.0 beforehand. The flex abrasion resistance was essentially unchanged, remaining about five times as great as that of broadcloth woven of unmercerized yarn. The tearing strength likewise was unaffected, and was double that of the untreated control woven of unmercerized yarn. The results show that the benefits of yarn mercerization under high tension--enhanced luster, tearing strength, and abrasion resistance--are retained after the silicone-peroxide treatment, and a fair level of wash-wear performance is imparted.

The curing of silicone-peroxide emulsion on fabric in which creases have already been formed has been observed to increase moderately the durability of the creases to laundering. This effect may prove of value when silicone treatments are used in conjunction with current methods of imparting durable pleats.

DISCUSSION

Question: Have fabric treatments been carried out with the benzoyl peroxide, emulsifiers and solvents present in the treating bath but with the silicone absent?

C. M. Welch: Yes. Control experiments omitting the dimethyl silicone or omitting the peroxide have been run. In the absence of silicone, no appreciable increase in wrinkle recovery in either the wet or conditioned state was obtained. With silicone present but peroxide absent, a small increase (20-30°) in conditioned recovery but no increase whatever in wet recovery were noted. The results force us to the conclusion that the combination of silicone and the peroxide are essential to obtain large increases (90-110°) in wrinkle recovery.

Question: Were dimethyl silicones you used in these treatments end-blocked?

C. M. Welch: Yes. We used the standard dimethyl silicone oils and emulsions which are designed for applications requiring high heat stability.

Question: How were the treated fabrics laundered and dried in making wash-wear ratings?

C. M. Welch: They were laundered in an automatic washing machine at 140°F. and tumble dried.

Question: Would the temperatures used in washing affect the behavior of the finish?

C. M. Welch: Probably not very much, although we have not studied this aspect.

Question: Are the silicone-treated fabrics thermoplastic?

C. M. Welch: Most of the saturated organic polymers which we have crosslinked as films on cotton fabrics still show a degree of softening and thermoplasticity when heated, even though they are crosslinked to the extent of being immune to solvent extraction. However, the silicone finishes are already so soft at room temperature that it is hard to notice much effect on them at elevated temperature.

Question: Have you determined wash-wear ratings from drip-dried fabrics?

C. M. Welch: No. The use of drip drying should lead to higher ratings for silicone-finished fabrics than does tumble drying, since the fabrics are subjected to large deforming forces in tumble drying. We have not studied this experimentally, however.

Question: The wrinkle recovery values you obtained on silicone-treated fabrics were relatively high; yet only medium-to-fair wash-wear ratings were obtained. How do you account for this?

C. M. Welch: Quite possibly the weight applied to fabric in the Monsanto wrinkle recovery test is not as great as it should be to correspond adequately to the large deforming forces caused by tumble drying with a 4-pound load.

Question: Can higher wash-wear ratings be obtained by using higher add-ons?

C. M. Welch: Our limited data obtained at somewhat higher add-ons than those mentioned

here (8 to 13 percent) indicate that probably a plateau would be reached.

Question: Do these fabrics show increased wet soiling during laundering in the presence of other soiled fabrics?

C. M. Welch: We have not laundered silicone-treated fabrics in the presence of added soils. We have noticed that the fabrics pick up lint readily, due to static charge, and also take up certain dry soils. They are resistant, however, to certain kinds of staining, as with writing inks. At least one silicone is available commercially as a softener which is resistant to wet-soiling and imparts enhanced ease of soil removal.

Question: Have you used the maximum add-on of silicone possible?

C. M. Welch: There does not seem to be any maximum for the saturated organic polymers we have applied with peroxide-induced crosslinking. The add-on can be increased as much as desired by raising both the polymer and the peroxide levels.

Question: Have you tried combining conventional cellulose crosslinking agents with silicone treatments in order to get the highest wash-wear ratings?

C. M. Welch: We have done this in two ways: by applying first the cellulose crosslinking agent and then the silicone-peroxide formulation, and also by applying the combination in a single bath. Although we have not as yet been satisfied with the improvement in abrasion resistance obtained by these combination treatments, the amount of cellulose crosslinking needed to obtain high wrinkle resistance has been greatly decreased.

Question: Have you measured the moisture absorption of the silicone-treated fabrics?

C. M. Welch: This question is of interest in establishing the machanism of the creaseproofing effect we have obtained. Previous workers have found that silicone-type finishes do decrease the dynamic moisture absorption of cotton. Conner and Reeves observed this on "silicone alloy"-treated fabrics. However, I feel the primary cause for the wrinkle resistance and wash-wear properties given by crosslinked silicone films is simply the enormous resiliency of such films.

PHYSICAL AND CHEMICAL MODIFICATION TO IMPROVE ABRASION RESITANCE OF CROSSLINKED COTTONS

[Summary]
by
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(Presented by A. S. Cooper, Jr.)

The rapid deterioration of durably pressed all cotton garments is generally accepted to be caused by the reduction in the physical properties when cotton cellulose is crosslinked to the extent necessary for no-iron drying characteristics and sharp crease retention after washing. One method of possibly reducing the effects of the cellulosic crosslinks is to preferentially select regions of the fabric structure for crosslinking the cellulose. This, for example, permits the exposure of untreated cotton in regions of maximum wear. Another approach for increasing the toughness of the fabric is to improve yarn and fabric elongation. Combinations of these approaches have resulted in a significant improvement in the wear resistance of durably pressed cotton fabrics.

Cotton fabrics with no pretreatment and the same fabrics pretreated by slack mercerization with and without controlled restretching techniques were preferentially crosslinked on the back side of the fabric. Preferential crosslinking was accomplished either by backcoating the fabric with a viscous solution of the crosslinking agent or by face coating a previously resin impregnated fabric with a viscous solution of a catalyst inactivating agent. Laboratory abrasion tests showed up to twentyfold increases in flex abrasion resistance and up to threefold increases in flat abrasion resistance. Washing and tumble drying tests of pant cuffs formed by delayed curing showed a significant improvement in resistance to wear after 20 cycles. However, all of the samples were damaged with the extent of the abrasion being less for the preferentially crosslinked samples than that for the conventionally crosslinked samples. Slack mercerization followed by partial restretching slightly improved wear resistance in comparison with the other pretreatments.

Since a technique for producing durably creased garments with a cotton face did not completely eliminate wearing damage, a series of washing and tumble drying tests were made on untreated cotton cuffs with the seams stitched in to simulate the crease retention characteristics of durably pressed cuffs. After only one wash and drip dry cycle there was noticeable abrasive damage at the cuff tip. After five washing and tumble drying cycles a small hole had been worn in the tips of the cuffs. After 20 wash-tumble dry cycles abrasive damage was extensive. On the basis of these results there is strong indication that untreated cotton does not have sufficient abrasion resistance to withstand the combined effects of washing and tumble drying if the cotton fabric is set to cause the identical area to abrade each time. These results confirm those obtained with durably pressed cuffs that were preferentially crosslinked to give an essentially untreated cotton on the abrading surface. To obtain satisfactory wear life in durably pressed garments, the resistance to wear at sharp creases apparently must be better than that exhibited by untreated cotton which is mechanically held in a creased condition.

The application of conventional softening and lubricating agents to the untreated cotton surface caused an improvement in abrasion resistance, but the agents are gradually removed by repeated washing and tumble drying. The application of polymer forming material to the untreated cotton surface caused an improvement in dye retention with little change in abrasion resistance as determined by washing and tumble

drying. Optimum formulations have not been determined, but the most encouraging results have been obtained by face coating the cotton fabric with formulations containing a polymer forming resin and suitable modifiers then drying and curing the fabric. The fabric is then back coated with a resin formulation for delayed curing. The types of polymer forming resins that seem to offer the most promise are those that polymerize easily without extensive reaction with the cotton cellulose. Examples are APO-methylol melamine, methylated methylol melamine, THPC-methylol melamine, and THPC-APO. The results of washing tests of samples coated on a laboratory coater indicate that durably creased cuffs may be produced to withstand 5 washing and tumble drying cycles without change in appearance and 20 cycles with only wear, but no holes. These results were obtained in washing and drying machines which abrade cotton goods at an excessive rate.

These results show a definite improvement in comparison with conventionally delayed cure cuffs on the basis of evaluation by washing and tumble drying. It is recognized that this type test does not necessarily mean that comparable improvements will be obtained under actual service conditions. Service tests will, of course, be made as soon as possible to determine the actual value of these preferential crosslinking treatments.

DISCUSSION

Question: Did you use colloidal silica in your formulation and did this improve resistance to laundering and tumble drying?

A. S. Cooper, Jr.: Yes. It reduced interfiber friction. We also used zirconium oxide with similar results.

Question: Have you tried polyurethanes for coatings?

A. S. Cooper, Jr.: No, but we plan to.

BLENDS OF CHEMICALLY TREATED AND UNTREATED COTTON FIBERS TO IMPROVE ABRASION RESISTANCE

[Summary]
by
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(Presented by R. J. Cheatham and N. B. Knoepfler)

Crosslinking resins have long been known to significantly lower the abrasion resistance and strength, particularly tearing strength, of cotton textile products. The recent evolution of the so-called "permanent press" process has accentuated the problem of abrasion resistance, because higher resin add-ons are being used to obtain better levels of appearance. Moreover, the use of higher curing temperatures to speed up production is thought to have further accentuated this problem. It was found that resinated cotton products were degraded to the point where significant abrasion damage resulted in fewer than 10 cycles of washing and tumble dryings. In an effort to overcome this deficiency in abrasion resistance, the textile industry has resorted to blending cotton with polyester and nylon 420 fibers. This approach suggests that blends of chemically treated and untreated cotton fibers might give improved abrasion resistance and make cotton products more competitive in this large market.

Since previous experience with the resination of unbleached cotton yarns had shown that it was difficult to obtain uniform resin impregnation, two bales of 1-1/16" cotton of average strength (82,000 pounds Pressley) and fineness (4.0 Micronaire reading) was scoured and bleached for experimental use.

Initial experiments were conducted with blends of resinated and unresinated cotton fibers in the form of filling in a standard 8 harness filling sateen having 64 ends and 112 picks per inch using 30/1 untreated warp and 30/1 filling yarns made from varying percentages of resinated and unresinated cotton fibers. Trouser cuffs made from these fabrics when pressed and cured showed promising results with respect to resistance to abrasion when subjected to repeated washing and drying cycles in the type of home laundry equipment recommended by the American Association of Textile Chemists and Colorists for evaluating the wash-wear characteristics of permanent press garments. The cuffs containing the highest resin add-ons showed the greatest wear, and those containing the lowest resin add-ons lasted through 60 washing and drying cycles.

These promising results encouraged us to experiment with blends of varying proportions of resinated and unresinated fibers in both warp and filling in a twill structure similar to those now used in men's and boy's slacks. A 3-up 2-down steep twill having 126 ends of 50/2 and 50 picks of 13.5/1 per inch, weighing 6.15 ounces per square yard was used for this evaluation. The resin treated fibers were blended at the picker to obtain the 50/50 and 75/25 percent blends and carded at approximately 10 pounds per hour on a metallic clothed card. The 25/75 blend was obtained at the first drawing process. The warp yarns were made from the 50/50 percent blends only. No particular trouble was experienced in processing the yarns containing the blends or the yarns for the control fabric made from the scoured and bleached cottons. End breakage for the 50/1 yarn made from the 50/50 percent blend was 44.7 as compared with 19.7 EDPTSH (ends down per thousand spindle hours) for the scoured and bleached cotton. However, 1-1/16" cotton is shorter than usually used for spinning 50/1 yarn. No end breakage

occurred while spinning the 13.5/1 control filling and the short yardages of 13.5/1 filling yarns from the blends of resinated and unresinated cotton. The tearing strength and resistance to flex abrasion of the fabrics made from the blends of resinated and unresinated cottons were higher in all cases than the control fabrics conventionally treated to about the same resin contents as the blended fabrics. However, the reverse is true for the flat abrasion values. As was expected, the control fabrics conventionally resinated where all the fibers were resinated had higher crease angles than the fabrics composed of blends of resinated and unresinated fibers.

Trouser cuffs made from the 50/50 blend of resinated and unresinated cotton fibers in both warp and filling averaged 22 washing and drying cycles before a defect (one or more broken threads) due to abrasion occurred as compared with 11 cycles for cuffs made from the control fabric conventionally resinated, and 6 and 7 cycles for cuffs made from conventionally resinated commercial 7 ounce twill and 8 ounce sateen, respectively. Although the trouser cuffs made from the 50/50 percent blend of resinated and unresinated cotton in both warp and filling had several minor defects after 40 washing and drying cycles, such as small holes (one or more broken threads) and a slight pucker inside the bottom of the cuff, they were still considered serviceable for casual wear.

Comparisons of the effect of different curing times and resin contents on wear life of the control fabrics and fabrics composed of blended fibers are also discussed.

Although the results obtained to date indicate that significant improvements in abrasion resistance of "permanent press" products can be obtained through the use of blends of resinated and unresinated cotton, the research is still too limited to draw hard and fast conclusions.

DISCUSSION

Question: Did puckering occur in your samples, and if so where did it take place? Also, was there less puckering when a higher percent treated fiber was present?

N. B. Knoepfler: Yes, some puckering did occur. Slight puckering occurred in the seam when the samples were cut in the filling direction, and in the cuff when cut in the warp direction. However, all of the puckering was very slight.

Question: How do you plan to eliminate shrinkage of the untreated fibers?

R. J. Cheatham: This would best be done with a study of the yarn and fabric structure. We expect to do this in future work.

Question: Where did the fiber loss occur--warp or filling?

R. J. Cheatham: We found many different types of abrasive damage. However, we do not have enough data to date to fully answer that question. Most abrasive damage presently occurs in the warp yarns only because most pants are cut in the warp direction.

Question: Was drawing frame blending tried?

R. J. Cheatham: Yes. The 25 percent treated--75 percent untreated sample was blended at the drawing frame.

Question: What about card waste?

R. J. Cheatham: We did not find excessive fiber breakage or waste at the card. Even drafting of the fibers was evident. This was helped by the fact that a softener was put on the bleached and scoured rawstock. The softener used was TMN (alkylaryl polyglycol).

Question: Was yarn crimp greater in the warp or filling?

R. J. Cheatham: We have not yet measured the yarn crimp. We realize that a balanced crimp would be helpful.

Question: Wouldn't a more realistic evaluation be based on equal crease recovery angles?

N. B. Knoepfler: True, but one doesn't know what the crease angles will be until after the samples are made. We now have data to work with which may make such an evaluation possible in the future.

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Arthur, Jett C., Jr. - Cotton Chem. Reactions Lab.

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